

UNITED STATES DISTRICT COURT IN CLERK'S OFFICE
EASTERN DISTRICT OF NEW YORK DISTRICT COURT E.D.N.Y.

FILED
★ DEC 16 2005 ★

PALL CORPORATION,

Plaintiff,

v.

ENTEGRIS, INC., d.b.a.

MYKROLIS CORPORATION,

Defendant.

LONG ISLAND OFFICE

Civil Action No. _____

CV-05 5894

(Jury Trial Demanded)

SEYBERT, J.

COMPLAINT

Plaintiff Pall Corporation ("Pall" or "Plaintiff"), by and through its attorneys, for its complaint against Defendant Entegris, Inc., d.b.a. Mykrolis Corporation ("Defendant"), alleges as follows:

PARTIES

1. Pall is a corporation organized and existing under the laws of the state of New York with a principal place of business at 2200 Northern Boulevard, East Hills, New York 11548.

2. Upon information and belief, Entegris, Inc. is a corporation organized and existing under the laws of Minnesota with a principal place of business at 3500 Lyman Blvd., Chaska, Minnesota 55318.

3. Upon information and belief, on or about August 6, 2005, Entegris, Inc. completed its merger with Mykrolis Corporation. Upon information and belief, Mykrolis Corporation operates as a "Doing Business As" entity of Entegris, Inc. Prior to the merger, upon information and belief, Mykrolis Corporation was a corporation organized and existing

under the laws of the state of Delaware with a principal place of business at 129 Concord Road, Billerica, Massachusetts 01821.

JURISDICTION AND VENUE

4. This action arises under the Patent Laws of the United States, 35 U.S.C. §§ 1 *et seq.* Subject matter jurisdiction is conferred by 28 U.S.C. §§ 1331 and 1338(a).

5. On information and belief, Defendant is and has been doing business, either directly or through its agents, in this judicial district and elsewhere in the United States. Personal jurisdiction over Defendant exists under New York CPLR §§ 301 and 302.

6. Venue in this judicial district is proper pursuant to 28 U.S.C. § 1400(b), as defined by 28 U.S.C. §§ 1391(b) and (c).

U.S. PATENT NO. 5,510,026

7. United States Patent No. 5,510,026 ("the '026 patent") entitled "Filter Arrangement Including A Non-Reentrant Shape" was issued by the United States Patent and Trademark Office ("PTO") on April 23, 1996. A copy of the '026 patent is attached hereto as Exhibit A.

8. Pall owns all right, title, and interest in the '026 patent.

9. The '026 patent remains in full force and effect.

U.S. PATENT NO. 6,174,439

10. United States Patent No. 6,174,439 ("the '439 patent") entitled "Filter Package" was issued by the PTO on January 16, 2001. A copy of the '439 patent is attached hereto as Exhibit B.

11. Pall owns all right, title, and interest in the '439 patent.

12. The '439 patent remains in full force and effect.

INFRINGEMENT OF THE '026 PATENT

13. On information and belief, Defendant has infringed the '026 patent directly, by contribution, and/or by inducement, in this district and elsewhere in the United States, by making, offering to sell, selling, and/or using products, such as its WAFERGARD® MAX product, that embody and/or employ the claimed inventions of the '026 patent. Upon information and belief, a true and accurate copy of a WAFERGARD® MAX product brochure is attached hereto as Exhibit C.

14. Defendant's infringement of the '026 patent has caused substantial injury to Pall, for which Pall is entitled to receive damages adequate to compensate it for such infringement.

15. On information and belief, Defendant's infringement of the '026 patent has been committed in a willful manner, and in deliberate and intentional disregard of Pall's rights.

INFRINGEMENT OF THE '439 PATENT

16. On information and belief, Defendant has infringed the '439 patent directly, by contribution, and/or by inducement, in this district and elsewhere in the United States, by making, offering to sell, selling, and/or using products, such as its QUICKCHANGE® ATM 0.05 micron product, that embody and/or employ the claimed inventions of the '439 patent. Upon information and belief, a true and accurate copy of Defendant's QUICKCHANGE® ATM product brochure is attached hereto as Exhibit D.

17. Defendant's infringement of the '439 patent has caused substantial injury to Pall, for which Pall is entitled to receive damages adequate to compensate it for such infringement.

18. On information and belief, Defendant's infringement of the '439 patent has been committed in a willful manner, and in deliberate and intentional disregard of Pall's rights.

DEMAND FOR JURY TRIAL

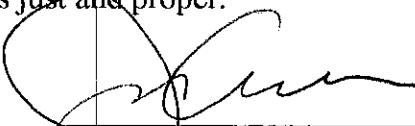
19. Pall demands a trial by jury to the extent permitted by applicable law.

DEMAND FOR RELIEF

WHEREFORE, Pall prays that this Court enter judgment in its favor and against Defendant and grant the following relief:

- A. Permanently enjoin Defendant, its employees and agents, and any others acting in concert with Defendant, from infringing the '026 patent and/or the '439 patent;
- B. Award Pall its damages resulting from Defendant's infringement of the '026 patent and/or the '439 patent;
- C. Award Pall treble damages pursuant to 35 U.S.C. § 284 as a result of Defendant's willful infringement of the '026 patent and/or the '439 patent;
- D. Declare this case exceptional pursuant to 35 U.S.C. § 285 and award Pall costs and attorney fees; and
- E. Grant Pall such other relief as is just and proper.

Date: December 16, 2005



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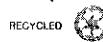


Exhibit A

US005510026A

United States Patent [19]

Geibel et al.

[11] Patent Number: **5,510,026**
 [45] Date of Patent: **Apr. 23, 1996**

[54] **FILTERING ARRANGEMENT INCLUDING A NON-REENTRANT SHAPE**

[75] Inventors: Stephen A. Geibel, Cortland; Scott D. Hopkins, Dryden; William L. Murphy, Homer; John D. Miller, Ithaca, all of N.Y.

[73] Assignee: Pall Corporation, East Hills, N.Y.

[21] Appl. No.: 408,191

[22] Filed: Mar. 22, 1995

Related U.S. Application Data

[63] Continuation of Ser. No. 888,206, May 26, 1992, abandoned.

[51] Int. Cl.⁶ B01D 27/08

[52] U.S. Cl. 210/232; 210/455; 210/497

[58] Field of Search 210/232, 236, 210/238, 435, 451, 455, 460, 497.01, 497.2

[56] References Cited**U.S. PATENT DOCUMENTS**

4,170,556 10/1979 Pall ,

4,295,525	10/1981	Swift, Jr. et al. .	
4,608,166	8/1986	Cain	210/232
4,623,456	11/1986	Ratner	210/232
4,865,727	9/1989	Krauss	210/238
4,961,850	10/1990	Combest	210/232
5,035,797	7/1991	Janik	210/232

FOREIGN PATENT DOCUMENTS

2209593	7/1974	France	B01D 27/00
1297651	11/1969	United Kingdom .	
2075364	11/1981	United Kingdom	B01D 27/08

Primary Examiner—Robert A. Dawson

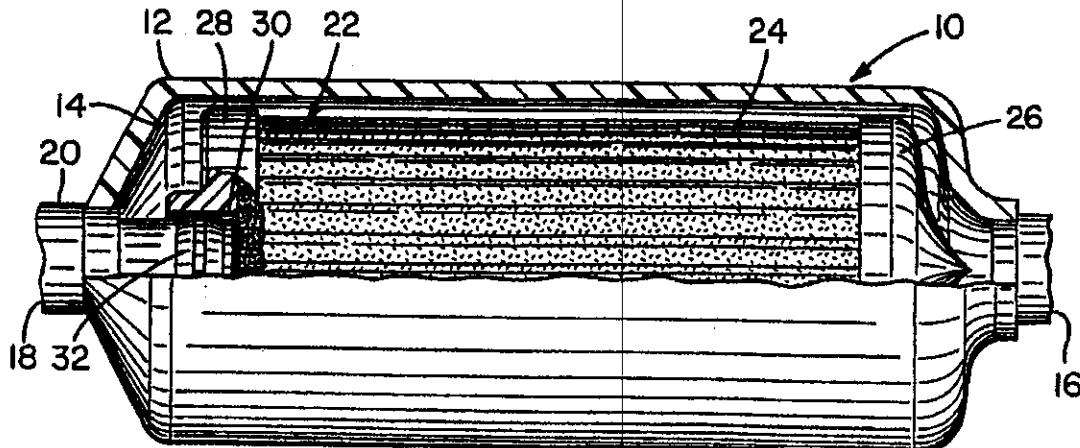
Assistant Examiner—W. L. Walker

Attorney, Agent, or Firm—Leydig, Voit & Mayer

[57] ABSTRACT

The disclosure describes a filter arrangement comprising a fitting and a filter assembly. The filter assembly includes an end cap and the end cap has a wall defining a bore. The fitting and the wall of the bore are fitted to one another by thermal expansion and contraction under elastic stress. At least one of the fitting and the wall of the bore has a non-reentrant shape shaped to resist longitudinal movement on the fitting within the bore and longitudinally secure and seal the filter assembly to the fitting.

62 Claims, 3 Drawing Sheets



U.S. Patent

Apr. 23, 1996

Sheet 1 of 3

5,510,026

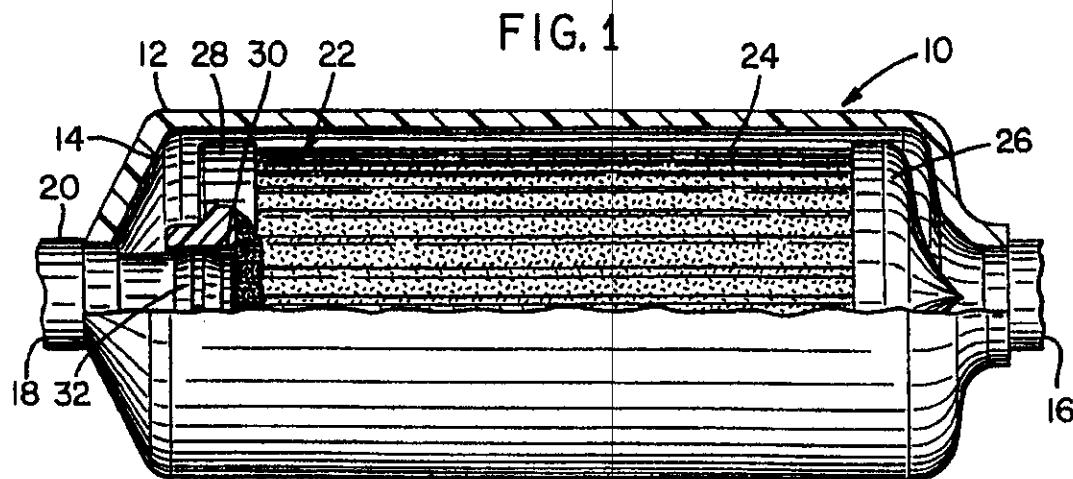
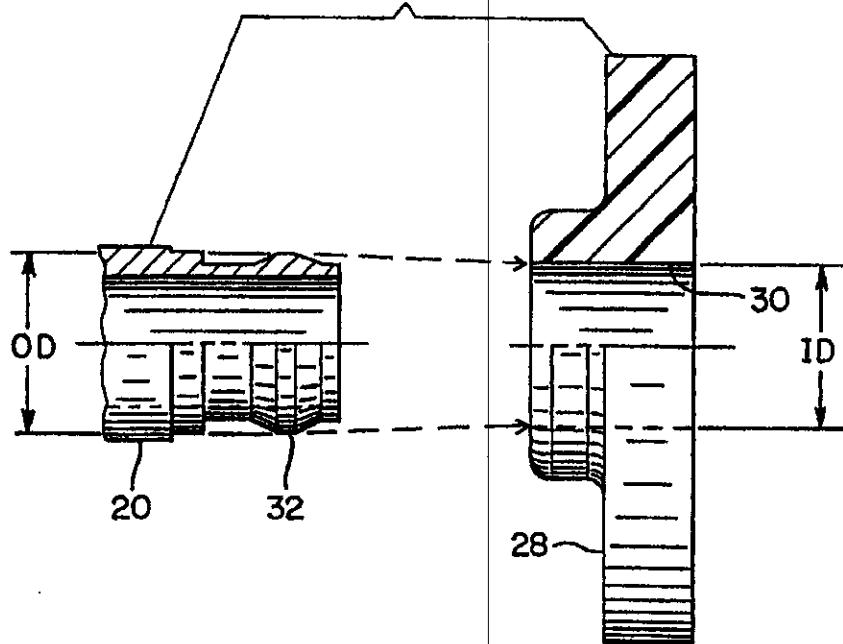


FIG. 2



U.S. Patent

Apr. 23, 1996

Sheet 2 of 3

5,510,026

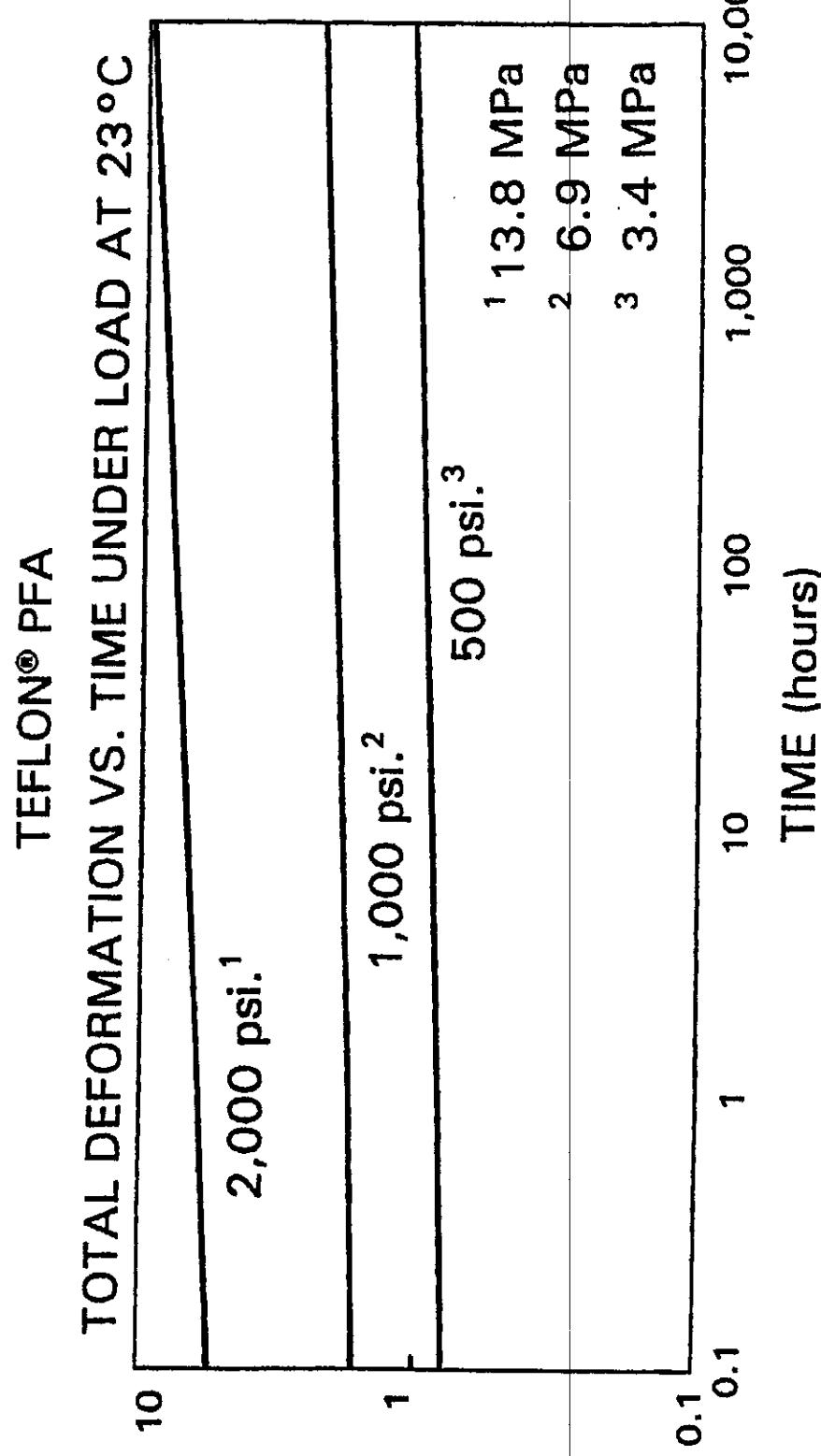


FIG. 3

U.S. Patent

Apr. 23, 1996

Sheet 3 of 3

5,510,026

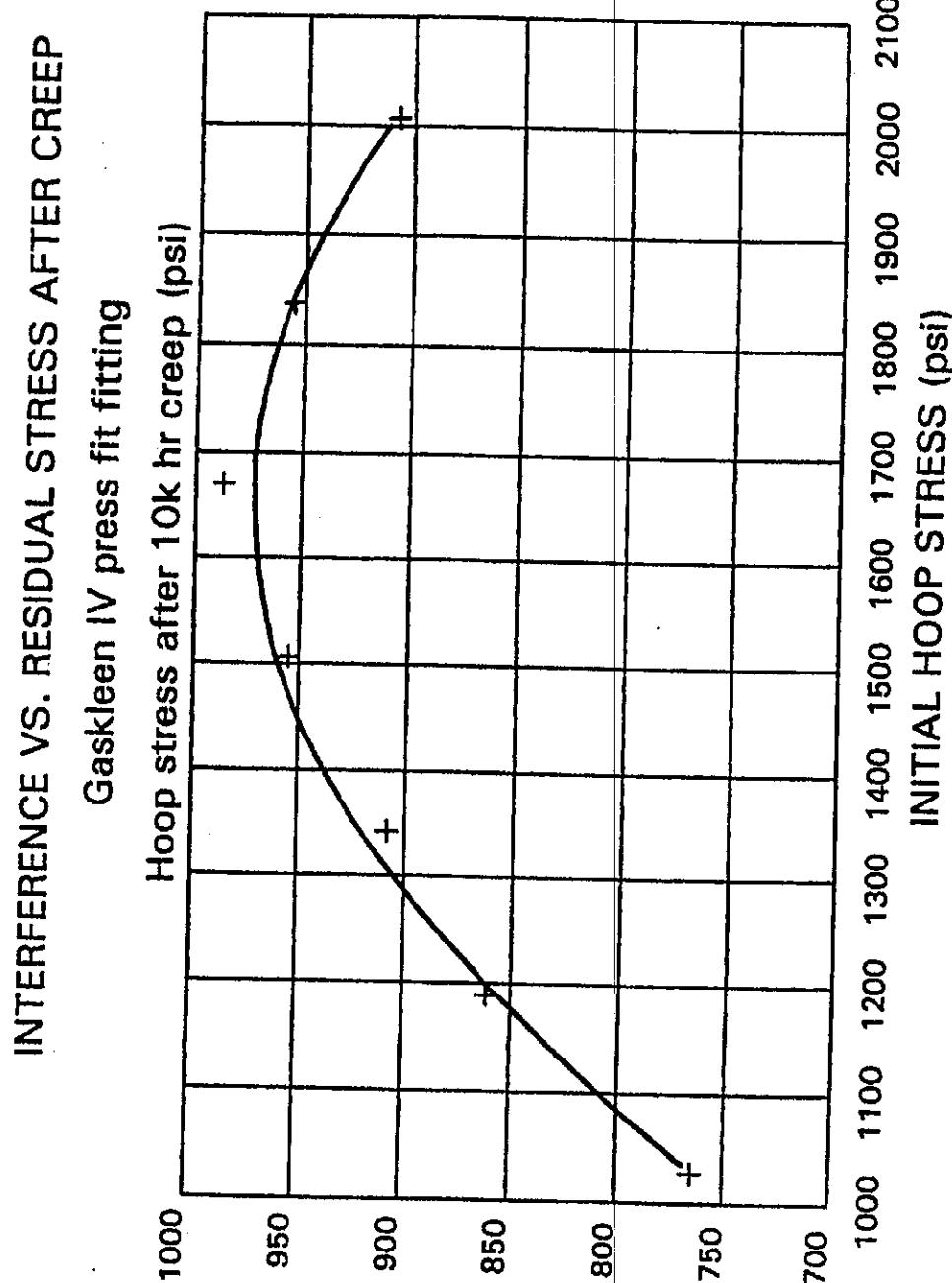


FIG. 4

5,510,026

1

FILTERING ARRANGEMENT INCLUDING A NON-REENTRANT SHAPE

This application is a continuation of application Ser. No. 07/888,206, filed May 26, 1992, now abandoned.

FIELD OF THE INVENTION

The invention relates generally to filter assemblies, and, more particularly, to methods and arrangements for providing a leak-tight seal between a filter cartridge and housing, and securing the filter cartridge to the housing.

BACKGROUND OF THE INVENTION

In filter assemblies, a filter cartridge is generally disposed within and secured to a housing. The filter cartridge is often secured to the housing by separate components, such as tie rods, or flanges and bolts, or the like. These separate components, however, result in added inventory and equipment expenses, as well as assembly costs.

The filter cartridge is typically tightly sealed to the housing at its inlet or outlet to preclude the bypassing of fluid or contaminants. Replacement of the filter cartridge may be accomplished by replacing the entire filter assembly in a housing that has been permanently closed, for example, by welding. Alternately, in assemblies where the housing is not permanently sealed, the filter may be removed from the housing for separate replacement or disposal.

Filter cartridges generally comprise a filter material and one or more end caps. Filter cartridge end caps are frequently constructed of polymeric materials, while housings are generally metal or the like. The filter cartridge is often sealed to the housing by means of an separate elastomeric material disposed between mating surfaces of the end cap and the housing. Such elastomeric seals are typically flat or O-ring gaskets disposed between annular surfaces of the end cap and the housing.

A number of disadvantages are associated with arrangements which utilize extraneous securing devices or gaskets, such elastomeric seals. For example, in order to accommodate piston-type O-ring seals, grooves or complex crevices must generally be molded or machined in the annular surface of the end cap. Similarly, where extraneous securing devices are utilized, the housing and/or the filter cartridge must be molded or machined to accommodate the extraneous devices. Manufacture of these components may require more complex tooling or additional operations, and, therefore, results in additional costs. Further, crevices in such designs often provide a dead volume that may be undesirable for filtration of ultrapure gas systems.

Additionally, elastomeric seals, and piston-type O-ring seals in particular, have a relatively low resistance to relative longitudinal motion of the mating parts. Relative motion between components can chafe the components and generate debris within the flow path. In more extreme situations, the filter may be forced off the mating housing fitting by reverse flow, or system "hammer." Consequently, the filter assembly often includes additional means for preventing longitudinal movement of the filter cartridge within the housing, as, for example, by a mechanical interlock.

Further, elastomeric materials are often subject to outgassing under certain conditions. This phenomenon is particularly troublesome in high purity gas filtration, such as in the manufacture of electronic chips. In chip manufacture, the filter elements are typically constructed entirely of

2

TEFLON® PFA. Consequently, it may be difficult to provide an effective seal between the filter end cap and the housing. Moreover, elastomeric seals, as well as extraneous securing devices, may be a source of contamination and potential chemical incompatibility.

OBJECTS OF THE INVENTION

It is a primary object of the invention to provide a filter assembly arrangement in which a filter end cap is effectively sealed to a housing without the use of a gasket. More particularly, it is an object of the invention to provide an effective sealing arrangement that alleviates the disadvantages incumbent with the use of such seals.

Another primary object is to secure a filter assembly to a housing without the use of extraneous structures.

Another object is to provide filter assembly that includes a minimal number of components and may be easily and economically manufactured and assembled.

An additional object is to provide a sealing and securing arrangement having improved structural integrity. A related object is to provide a sealing arrangement that is resistant to relative longitudinal motion of mating components, and does not require the use of external supports, such as mechanical interlocks, to prevent relative longitudinal motion. A related object is to provide an arrangement that seals and secures a filter assembly to a housing.

Yet another object is to provide a sealing arrangement that minimizes the compatibility considerations between the construction materials and the fluids to be filtered. A further object is to provide a filter arrangement that does not introduce additional sources of contamination, such as those introduced by out-gassing of elastomeric O-rings or gases retained in the dead volume presented by grooves provided for O-rings.

BRIEF SUMMARY OF THE INVENTION

In accomplishing these and other objectives of the invention, there is provided a filter assembly arrangement wherein an end cap engages a fitting as a result of thermal expansion and contraction, effectively sealing and securing the end cap to the fitting.

In accordance with the one aspect of the invention, a filter arrangement comprises a fitting and a filter assembly. The filter assembly includes an end cap and the end cap has a wall defining a bore. The fitting and the wall of the bore are fitted to one another by thermal expansion and contraction under elastic stress. At least one of the fitting and the wall of the bore has a non-reentrant shape shaped to resist axial movement of the fitting within the bore and axially secure and seal the filter assembly to the fitting.

In accordance with another aspect of the invention, a filter arrangement comprises a fitting and a filter assembly mounted to the fitting. The fitting includes a portion having a non-reentrant shape. The filter assembly includes a filter and an end cap mounted to the filter and having a wall which defines a bore. The end cap is expandable to a first size at a first predetermined temperature wherein the bore is large enough to accept the non-reentrant portion of the fitting, and the end cap is contractible to a second size at a second predetermined temperature wherein the wall of the end cap is sealed tightly about the non-reentrant portion of the fitting.

In accordance with a further aspect of the invention, a filter arrangement comprises a housing assembly and a filter assembly. The housing assembly includes a housing cham-

5,510,026

3

ber having an aperture and a hollow fitting extending from the aperture into the housing chamber. The hollow fitting has an outer surface with a non-reentrant shape. The filter assembly is disposed in the housing chamber and includes a filter material and an end cap connected to the filter material. The end cap has a bore with a wall. The wall is sealed tightly about the outer surface of the fitting by an interference fit at ambient temperatures, and the end cap and the fitting are joined to one another by thermal expansion and contraction of at least one of the end cap and fitting.

In accordance with a still further aspect of the invention, a filter assembly arrangement comprises a housing assembly and a filter assembly. The housing assembly includes a housing chamber having at least two apertures defining a flow path through the chamber. The housing assembly further includes at least one extension disposed within the housing chamber at one of the apertures, and the extension has an internal bore through which flow proceeds. The extension also has a non-reentrant shaped portion which extends into the housing chamber, and a non-reentrant shaped portion of the extension is defined by an outer diameter. The filter assembly comprises a filter material and at least one end cap. The end cap includes a substantially annularly shaped bore defined by a wall having an inner diameter. The inner diameter of the bore wall is smaller than the outer diameter of the non-reentrant shaped portion of the extension at ambient temperatures when the non-reentrant shaped portion is not disposed within the end cap bore. The filter assembly is disposed within the housing chamber in the flow path, and the non-reentrant shaped portion is disposed within the end cap bore during use. The extension and the end cap are joined to one another by thermal expansion and contraction such that there is an interference between the inner diameter and the outer diameter that induces stress in the end cap. The end cap bore and the non-reentrant shaped portion of the extension are sealed to one another under elastic stress at ambient temperatures so that the end cap fits tightly to the non-reentrant shaped portion without the presence of a sealing member.

In order to assemble the end cap and the housing extension, either the end cap is heated to expand the bore, or the housing extension is cooled to contract and reduce the outer diameter of the extension. The components are then assembled and returned to room temperature or ambient temperature to provide a seal as a result of the interference contact between the components. By calculating the theoretical stress and strain induced in the end cap and/or the housing extension, both initially and following a given creep time, the optimal dimensions of the end cap and the extension may be selected to provide maximum sealing force between the components after creep by selecting components with dimensions that provide the maximum residual stress in the end cap and/or the housing extension after creep.

The filter assembly arrangement does not require the use of a gasket to seal the filter cartridge to the housing. Further, the arrangement does not generally require the use of extraneous structures for securing the filter cartridge to the housing. (It will be appreciated, however, that it may be desirable to support the weight of an extremely large or heavy filter cartridge to prevent undue stress on the seal.) Thus, the simplified arrangement minimizes the number of components required and simplifies the design of the components themselves, which reduces both manufacturing and assembly costs. For example, by the elimination of a gasket, the end cap and extension may be more easily fabricated, as no complex finishing operations are required. Similarly, as

4

the arrangement does not necessitate the use of extraneous support structures, the components do not require complex provisions for securing the filter cartridge to the housing. Consequently, the tooling required is greatly simplified and manufacturing costs reduced.

Further, the arrangement alleviates many disadvantages associated with the use of extraneous support structures, elastomeric seals or the like. In reducing the number of materials utilized in the arrangement, compatibility considerations between filter assembly materials and the fluids to be filtered are minimized. Further, sources of contamination, such as those introduced by the out-gassing of elastomeric seals or by gasses retained in the grooves provided for elastomeric seals, are likewise minimized.

Additionally, the filter assembly arrangement exhibits improved structural integrity of the seal itself. As the assembly has high resistance to relative movement between mating components, the sealing arrangement does not require the use of external supports to prevent relative longitudinal motion. Thus, the arrangement both seals and secures the filter assembly to the housing.

These and other features and advantages of the invention will be more readily apparent upon reading the following description of a preferred exemplified embodiment of the invention and upon reference to the accompanying drawings wherein:

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a plan view of a filter arrangement embodying the teachings of the invention. The filter arrangement is partially broken away to show the filter assembly.

FIG. 2 is an enlarged fragmentary view of the filter arrangement of FIG. 1 cut away and exploded to show the fitting and end cap.

FIG. 3 is a diagram of the total deformation over time of a TEFILON® PFA fluorocarbon resin under load at 73° F. (23° C.).

FIG. 4 is a diagram of the initial stress versus the residual stress after 10,000 hour creep for a representative end cap and fitting combination.

While the invention will be described in connection with certain preferred embodiments, there is no intent to limit it to those embodiments. On the contrary, the intent is to cover all alternatives, modifications, and equivalents included within the spirit and scope of the invention as defined by the appended claims.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Turning now to the drawings, there is shown in FIG. 1 a filter arrangement 10 comprising a housing 12, which includes a hollow housing chamber 14 with flow apertures 16, 18 that provide an inlet and an outlet through which fluid may flow into and out of the housing 12 to establish a flow path through the housing 12. (Although the following description refers to fluid flowing through the filter arrangement, the term "fluid" is intended to encompass both liquids and gases, or combinations thereof.) While the housing 12 illustrated incorporates the flow apertures 16, 18 at either end of the housing chamber 14, it will be appreciated that the flow apertures might alternately be provided at different positions within the housing.

The housing 12 further comprises a extension that extends into the housing chamber 14. In the embodiment shown, the extension is in the form of a fitting, which may be sealed to the housing 12 at one flow aperture 18 by any appropriate means. It will be appreciated, however, that the extension could alternately be in the form of an extension of the housing material itself, which protrudes into the housing chamber. While the fitting 20 could be disposed at either the inlet or outlet end of the housing 12, for the purposes of explanation, the filter arrangement 10 will be described as though the fitting 20 is disposed at the outlet end of the housing 12. Moreover, although the embodiment illustrated includes only one fitting 20, it will be appreciated that the arrangement could include fittings disposed within the flow apertures at both the inlet and outlet ends.

The fitting 20 may be constructed from a variety of metal materials including alloys, various metals, and combinations of metals and metal alloys. For example, nickel/chromium alloys, such as the Hastelloys, the Monels, and the Inconels are preferred. Of these, due to their corrosion-resistant properties, stainless steel alloys are preferred fitting materials. Similarly, however, metallic materials such as aluminum, magnesium, or the like, may likewise be suitable. It will further be appreciated that the fitting 20 could likewise be constructed of an alternate appropriate non-metallic material.

In order to filter fluid flowing through the housing 12, a filter cartridge 22 is disposed in the flow path within the housing 12. The filter cartridge 22 generally comprises a filter medium 24 to which one or more end caps 26, 28 are coupled. The filter medium 24 may comprise a porous membrane, or a woven or non-woven fibrous material, or other suitable material. While the end caps 26, 28 are generally fabricated of a polymeric material such as a fluorocarbon resin, it will be appreciated that they may be constructed from an alternate material, which exhibits desirable expansion and temperature characteristics. The filter cartridge 22 illustrated includes a cylindrical pleated filter 24 to which are coupled a blind end cap 26, and an end cap 28 having a bore 30. In this embodiment, the fluid flowing through the housing 12 is filtered as it flows outside in through the filter 24 and then the bore 30 or through the bore and inside out through the filter 24.

It will be appreciated that the filter cartridge and its components may be of alternative designs. For example, the filter may be of a cylindrical non-pleated design, or stacked thin plate design. Further, the cartridge may include bores in both end caps with the filter disposed therebetween so that fluid flows through one bore, the filter, and out the other bore. Alternatively, the filter may be of a "can" type design, and the cartridge may include only one end cap having a bore.

In order to ensure that fluid is purified by flowing through the filter 24, at least one end cap 28 is sealed to the housing 12. This is generally accomplished by sealing the substantially annularly shaped bore 30 of the end cap 28 to a substantially annularly shaped portion or end 32 of the fitting 20 that extends into the housing chamber 14.

In accordance with the invention, there is provided a filter arrangement 10 and a method of securing and sealing an end cap 28 to a housing fitting 20. The inventive sealing arrangement does not require the use of additional sealing materials such as an O-ring or flat ring gasket in order to provide an effective seal between the components. Rather, the internal diameter of the bore 30 of the end cap 28 (ID, shown in FIG. 2) is smaller than the outer diameter of the end 32 of the

fitting 20 (OD, shown in FIG. 2) to present an interference fit at ambient temperatures. The end cap 28 is installed on the fitting 20 by heating the end cap 28 to a first predetermined temperature to expand the internal diameter (ID) of the bore 30, and/or cooling the fitting 20 to reduce its outer diameter (OD) and slipping the parts together. (In the example that follows, an end cap fabricated from a TEFILON® PFA material and heated to 525° F. to expand the internal diameter.) The assembly is then permitted to return to a second predetermined temperature, such as ambient temperature. As the end cap 28 cools and the bore 30 contracts and/or the male fitting 20 heats and expands, intimate thermal contact is established between the surfaces of the end cap bore 30 and the housing fitting 20 to maintain the components in sealing engagement.

While the end 32 of the fitting 20 that extends into the housing chamber 14 and the bore 30 of the end cap 28 have been described as preferably substantially annularly shaped, it will be appreciated that the end 32 and the bore 30 may be of an alternate shape, so long as they may be assembled to provide an interference fit, which provides a stable seal between the components.

In a currently preferred embodiment of the invention, the end 32 is of a non-reentrant shape, i.e., the end 32 itself is shaped to resist movement once assembled into the bore 30. It will thus be appreciated that the shape of the end 32 itself increases the effectiveness of the seal and increases the resistance of the filter cartridge 22 to longitudinal movement relative to the housing 12. As shown in FIGS. 1 and 2, the OD of the end 32 includes an annular extension along that portion which is centrally disposed within the bore 30 once assembled. The annular surface of the end 32 bevels outward to a substantially flat annular surface, or to form a spherical section. This arrangement is particularly desirable because it may be easily machined. While the bore 30 may comprise a mating annular recess, the preferred embodiment of the invention includes a cylindrically shaped bore 30.

This method of sealing the components together, and the non-reentrant shape of the end 32 in particular, likewise assists in securing the filter cartridge 22 to the housing 12. Generally, no additional securing devices, such as tie rods or the like, will be required to further secure the cartridge 22 to the housing 12. It will be appreciated, however, that in filter assembly designs requiring the use of relatively large or heavy filter cartridges, it may be desirable to provide additional support for the cartridge at the end opposite the fitting 20. Such additional support may likewise be desirable when the filter assembly is utilized in environments with large range of pressure transients, for example in applications where the filter assembly will experience excessive hammer or shock. Such support may be provided by simple dimples or the like in the housing which protrude into the housing chamber and contact the filter cartridge 22.

According to an important aspect of the invention, the end cap 28 and the end 32 are dimensioned such that the elastic stresses that remain in the components as a result of the interference fit are optimal throughout the life of the assembly 10. In order to determine the optimal initial dimension of the end cap bore 30 relative to the end 32, the initial and residual stress and strain induced in the end cap 28 and/or the end 32 the fitting 20 as a is close to the interference must be considered. For our poses of explanation, only the stress and strain induced in the end cap will be considered because the modulus of elasticity of the end cap 28 is typically much greater than that of the fitting 20. Where the modulus of elasticity of the fitting 20 is close to or greater than that of the end cap 28, then the stress and strain

5,510,026

7

induced in both the fitting 20 and the end cap 28 may be considered and analogous considerations apply.

Representative sizes of an end cap fabricated from the currently preferred TEFILON® PFA material and a stainless steel fitting will be used as an example. TEFILON® PFA is currently preferred because of its desirable high temperature properties; TEFILON® PFA can withstand relatively high temperatures without melting. It will be appreciated, however, that other polymeric materials, such as polypropylenes or polyesters could likewise be utilized. Thus, there is no intention to limit the invention to the specific materials mentioned or to the specific dimensions mentioned. Further, it is expected that the arrangement would be equally applicable to fitting and/or end cap bores on the order of $\frac{1}{4}$ to $2\frac{1}{2}$ inches in diameter, or higher.

The following table summarizes calculations for the stress and strain induced in an end cap as result of different interference fits for several combinations of end cap ID and fitting OD. The table shows the initial stress and strain, as well as the residual stress and strain after 10,000 hours at room temperature.

DIMENSIONS		INITIAL		CREEP 10K Hrs. @ Room Temp.	
Fitting	End Cap	Strain (in./in.)	Stress (psi)	Strain (%)	Stress (psi)
O.D. (in.)	I.D. (in.)				
.299	.271	.103	1033	2.5	764
.301	.269	.119	1190	3	864
.303	.267	.135	1348	4	912
.305	.265	.151	1509	5	961
.307	.263	.167	1673	6.3	981
.309	.261	.184	1839	8	962
.311	.259	.201	2008	10	916

The initial stress and strain induced in the end cap may be determined with reference to the initial dimensions of the end cap and the dimensions of the end cap once installed on the fitting. The diameter strain (ϵ) is calculated according to the following equation, wherein ΔD is calculated by determining the change in the ID as a result of the interference fit, and D is the initial ID:

$$\epsilon = \Delta D / D$$

The hoop stress (σ) may then be calculated by multiplying the diameter strain by the modulus of elasticity (E) for the particular material utilized in the end cap.

$$E = \sigma / \epsilon$$

As indicated above, the currently preferred embodiment of the invention utilizes an end cap 28 fabricated from TEFILON® PFA. The modulus of elasticity for this material is 10,000 psi.

The resultant deformation of the end cap over time may be determined with reference to the creep data illustrated in FIG. 3. This figure shows the tensile strain observed under various loads at room temperature (i.e., 73° F., or 23° C.). The creep observed, described as a percent strain under a given load, may be determined by interpolating the logarithmic scale. For example, it may be seen that after 10,000 hours, the tensile strain for a hoop stress of 1033 psi is on the order of 2.5%.

The residual stress remaining in the end cap after 10,000 hours may then be determined using the natural dimensions of the end cap after creep strain. This determination involves

8

multi-step calculation. First, the natural dimensions of the end cap after 10,000 hours creep may be calculated according to the following equation:

$$5 \quad \text{initial ID} + (\text{initial ID}) (\% \text{ strain at 10,000 hours})$$

For example, for the end cap having an initial ID of 0.271 in., the diameter after 10,000 hours creep will be calculated as follows:

$$10 \quad 0.271 \text{ in.} + (0.271 \text{ in.})(2.5\%) = 0.278 \text{ in.}$$

Second, the diameter strain at 10,000 hours may be calculated as above using the new natural dimension of the end cap. Continuing with our example, for the end cap having an initial ID of 0.271 in., the diameter strain after 10,000 hours creep may be calculated as follows:

$$20 \quad (0.299 \text{ in.} - 0.278 \text{ in.}) / 0.278 \text{ in.} = 0.076 \text{ in./in.}$$

Third, the residual stress after 10,000 hours creep may likewise be calculated as above, i.e., by multiplying the hoop stress by the modulus of elasticity. Returning to our example, the residual stress may be calculated as follows:

$$25 \quad (0.076 \text{ in./in.})(10,000 \text{ psi}) = 764 \text{ psi}$$

In the examples in the above table, we have assumed that the assembly will be maintained at room temperature for a preponderance of the life of the assembly. It will be appreciated, however, that there is no intention to limit the assembly to room temperature use. The resultant deformation, and, consequently, the residual stress could likewise be determined with reference to time deformation curves for loads applied at alternate temperatures. Moreover, in the preferred embodiment, generally the polymeric female end cap has a higher coefficient of expansion than the metal male fitting. Consequently, when subjected to elevated temperatures, the female end cap will not be over stressed and permanently deformed.

40 The preferred fitting and end cap combination is considered to be the combination for the end cap that exhibits maximum residual stress over the life of the part (i.e., optimal hoop stress after 10,000 hours creep). The maximum residual hoop stress is graphically illustrated in FIG. 4.

45 The residual stress data for the representative end caps summarized in the table above are plotted as a function of initial stress. It will be appreciated that, generally, the dimensions for the fitting and end cap corresponding to the uppermost point of the curve will provide the optimal interference fit for residual stress. In this sample set of calculations, the end cap exhibiting the optimal residual stress after 10,000 hours at room temperature will be the end cap with an initial stress of 1673 psi, or the end cap with an inner diameter of 0.263 inches assembled on a fitting with an outer diameter of 0.037 inches.

55 Taking into account the practical considerations of manufacturing, the initial and residual stress and strain at the design tolerances may be calculated according to the methods explained above. The following table summarizes calculations for the stress and strain induced in the representative optimal dimension end cap (ID=0.262 - 0.003 in.) and fitting (OD=0.305 +/- 0.002 in.). Also provided in the following table are the dimensions of the inner diameter of the end cap after 10,000 hours at room temperature. It may be seen that the end cap will retain sufficient residual stress to provide an interference fit between the components and maintain the integrity of the seal.

5,510,026

9

10

DIMENSIONS		INITIAL		CREEP 10K Hrs. @ Room Temp.		
Fitting O.D. (in.)	End Cap I.D. (in.)	Strain (in./in.)	Stress (psi)	Strain (in./in.)	Stress (psi)	End Cap I.D. (in.)
.303	.265	.143	1434	4.5	942	.277
.307	.259	.185	1853	8	975	.280

Further, the assembly may be utilized at higher temperatures, while maintaining the integrity of the seal. As will be seen from the following dimensions, the end cap and fitting will maintain an interference fit after 10,000 hours at 392° F. (The dimensions of the components may be calculated as above.)

INITIAL DIMENSIONS		CREEP 10K Hrs. @ 392° F.	
Fitting O.D. (in.)	End Cap I.D. (in.)	Fitting O.D. (in.)	End Cap I.D. (in.)
.303	.265	.304	.285
.307	.259	.308	.288

Because the disclosed design does not utilize a gasket, its manufacturer does not require complex tooling or machining operations. Thus, those skilled in the art will appreciate that the described end cap and fitting may be more easily and economically manufactured than assemblies that include a gasket.

The practical considerations of assembly must likewise be taken into account in the design of the end cap and fitting. In order to ensure that the optimal end cap and fitting combination may be readily assembled, the dimensions of the end cap after heating may be calculated using the coefficient of thermal expansion (α) in in./in./°F., according to the following equation, wherein D is the initial diameter of the end cap in inches, Δ is the change in temperature in °F., and δ is the resultant change in diameter in inches:

$$\delta = \alpha D \Delta T$$

The coefficient of thermal expansion is relatively constant for a considerable range of temperatures. In general, the coefficient increases with an increase of temperature. For TEFLON® PFA material, the coefficient of thermal expansion is as follows:

70°-212° F.	$\alpha = 7.6 (10^{-5})$ in./in./°F.
212°-300° F.	$\alpha = 9.2 (10^{-5})$ in./in./°F.
300°-525° F.	$\alpha = 11.5 (10^{-5})$ in./in./°F.

(Alternate materials exhibit higher or lower coefficients of thermal expansion. For example, virgin homogeneous polypropylene has a coefficient of thermal expansion on the order of $5.0 (10^{-5})$ in./in./°F., while a polyester material has a coefficient of thermal expansion on the order of $3.3 - 5.2 (10^{-5})$ in./in./°F. It will be appreciated, however, that the temperature of the environment in which an end cap may be utilized will be limited by the coefficient of thermal expansion of the material from which the end cap is fabricated.)

The following reflects the dimensions of the representative end caps (at the design tolerance extremes identified above) at 525° F. While representative components in our example provide an interference fit during assembly, the end caps and fittings can be readily assembled.

DIMENSIONS	INITIAL	CREEP 10K Hrs. @ Room Temp.	Fitting O.D. (in.)	End Cap I.D. (in.)
Fitting O.D. (in.)	End Cap I.D. (in.)	Strain (in./in.)	Stress (psi)	End Cap I.D. (in.)
.303	.277	.303	.271	.271
.307	.271	.307	.277	.277

Further, it has been experimentally determined that the components may be readily assembled at significantly lower temperatures. As the end cap is heated, it becomes sufficiently soft to permit assembly to the fitting regardless of the interference. The following reflects the dimensions of the representative end caps (at the design tolerance extremes identified above) at 300° F. Although a residual interference exists to provide a snug fit, the components may be readily assembled.

DIMENSIONS	INITIAL	CREEP 10K Hrs. @ 392° F.	Fitting O.D. (in.)	End Cap I.D. (in.)
Fitting O.D. (in.)	End Cap I.D. (in.)	Fitting O.D. (in.)	End Cap I.D. (in.)	
.303	.270	.303	.264	.264
.307	.264	.307	.270	.270

In summary, the filter assembly arrangement and the method of sealing the filter end cap 28 to the housing 12 is accomplished as a result of thermal contraction of the end cap 28 and/or expansion of the end 32 of the fitting 20. The optimal dimension of the components may be calculated to provide maximum residual stress within the end cap 28 and/or the end 32 of the fitting 20 over the life of the filter cartridge 22. In utilizing a thermally engaged seal, the assembly does not require an additional gasket. As a result, the invention alleviates many of the disadvantages associated with the use of gaskets, such as elastomeric seals.

We claim as our invention:

1. A filter arrangement comprising a fitting including a portion having a non-reentrant shape and a filter assembly mounted to the fitting, the filter assembly including a filter and an end cap mounted to the filter and having an end cap wall defining a bore, the end cap being expandable to a first size at a first predetermined temperature wherein the bore is large enough to accept the non-reentrant portion of the fitting and being contractible to a second size at a second predetermined temperature wherein the wall of the end cap is sealed tightly about the non-reentrant portion of the fitting.

2. The filter arrangement of claim 1, wherein one of the end cap and the non-reentrant portion of the fitting is fabricated from a polymeric material and the other of the end cap and the non-reentrant portion of the fitting is fabricated from a metallic material.

3. The filter arrangement of claim 2, wherein the bore of the end cap has a cylindrical shape along an axis of the bore where the bore wall is sealed to the fitting.

4. The filter arrangement of claim 3, wherein the portion of the fitting having the non-reentrant shape extends outward to a substantially flat annular surface.

5. The filter arrangement of claim 4, further comprising a housing assembly including a housing chamber having an aperture communicating with the fitting, and wherein the filter assembly is disposed in the housing chamber in a flow path defined by the aperture.

6. The filter arrangement of claim 5, wherein the portion of the fitting having the non-reentrant shape has an outer diameter greater than the inner diameter of the bore of the end cap before the fitting is disposed in the bore, the end cap and the fitting being joined to one another by thermal

5,510,026

11

expansion and contraction of at least one of the end cap and the fitting to create a thermal interference fit for providing a leak tight seal between the filter assembly and the fitting and securing the filter assembly to the fitting.

7. The filter arrangement of claim 6, wherein the filter assembly further includes a blind end cap mounted to the filter.

8. The filter arrangement of claim 5, wherein a portion of the fitting has the non-reentrant shape and the non-reentrant portion extends outward to form a spherical surface.

9. The filter arrangement of claim 1, wherein at least one of the end cap and fitting is fabricated from a material that thermally expands and contracts.

10. The filter arrangement of claim 9, wherein the material includes a polymeric material.

11. The filter arrangement of claim 9 wherein the material includes a metallic material.

12. The filter arrangement of claim 1, further comprising a housing assembly including a housing chamber having an aperture communicating with the fitting, and wherein the filter assembly is disposed in the housing chamber in a flow path defined by the aperture.

13. The filter arrangement of claim 12, wherein the portion of the fitting having a non-reentrant shape has an outer diameter greater than the inner diameter of the bore of the end cap before the fitting is disposed in the bore, the end cap and the fitting being joined to one another by thermal expansion and contraction of at least one of the end cap and the fitting to create a thermal interference fit for providing a leak tight seal between the filter assembly and the fitting and securing the filter assembly to the fitting.

14. The filter arrangement of claim 1, wherein the bore of the end cap has a cylindrical shape along an axis of the bore where the bore wall is sealed to the fitting.

15. The filter arrangement of claim 1, wherein the bore wall comprises a mating annular recess which sealably engages the non-reentrant portion of the fitting.

16. The filter arrangement of claim 1, wherein the portion of the fitting having the non-reentrant shape extends outward to a substantially flat annular surface.

17. The filter arrangement of claim 1, wherein the portion of the fitting having the non-reentrant shape extends outward to form a spherical surface.

18. A filter arrangement comprising:

a fitting, and

a filter assembly including an end cap having a wall defining a bore, the fitting and the wall of the bore being fitted by thermal expansion and contraction under elastic stress to one another, wherein at least one of the fitting and the wall have a non-reentrant shape shaped to resist axial movement of the fitting within the bore and axially secure and seal the filter assembly to the fitting.

19. The filter arrangement of claim 18, wherein one of the end cap and the non-reentrant portion of the fitting is fabricated from a polymeric material and the other of the end cap and the non-reentrant portion of the fitting is fabricated from a metallic material.

20. The filter arrangement of claim 19, wherein the bore of the end cap has a cylindrical shape along an axis of the bore where the bore wall is sealed to the fitting.

21. The filter arrangement of claim 20, wherein a portion of the fitting has the non-reentrant shape and the non-reentrant portion extends outward to a substantially flat annular surface.

22. The filter arrangement of claim 21, further comprising a housing assembly including a housing chamber having an

12

aperture communicating with the fitting, and wherein the filter assembly is disposed in the housing chamber in a flow path defined by the aperture.

23. The filter arrangement of claim 22, wherein the fitting comprises a portion having the non-reentrant shape for engagement with the bore of the end cap and wherein the non-reentrant portion has an outer diameter greater than the inner diameter of the bore of the end cap before the end cap is fitted to the fitting, the end cap and the fitting creating a thermal interference fit for providing a leak tight seal between the filter assembly and the fitting and securing the filter assembly to the fitting.

24. The filter arrangement of claim 23, wherein the filter assembly further includes a blind end cap mounted to the filter.

25. The filter arrangement of claim 18, wherein at least one of the end cap and fitting is fabricated from a material that thermally expands and contracts.

26. The filter arrangement of claim 25, wherein the material includes a polymeric material.

27. The filter arrangement of claim 25, wherein the material includes a metallic material.

28. The filter arrangement of claim 18, further comprising a housing assembly including a housing chamber having an aperture communicating with the fitting, and wherein the filter assembly is disposed in the housing chamber in a flow path defined by the aperture.

29. The filter arrangement of claim 28, wherein the fitting comprises a portion having the non-reentrant shape for engagement with the bore of the end cap and wherein the non-reentrant portion has an outer diameter greater than the inner diameter of the bore of the end cap before the end cap is fitted to the fitting, the end cap and the fitting creating a thermal interference fit for providing a leak tight seal between the filter assembly and the fitting and securing the filter assembly to the fitting.

30. The filter arrangement of claim 18, wherein the bore of the end cap has a cylindrical shape along an axis of the bore where the bore wall is sealed to the fitting.

31. The filter arrangement of claim 18, wherein the bore wall comprises a mating annular recess which sealably engages the non-reentrant portion of the fitting.

32. The filter arrangement of claim 18, wherein a portion of the fitting has the non-reentrant shape and the non-reentrant portion extends outward to a substantially flat annular surface.

33. A filter assembly arrangement comprising, in combination:

a housing assembly comprising a housing chamber having at least two apertures defining a flow path there-through, the housing assembly further comprising at least one extension disposed within the housing chamber at one of the apertures, the extension having a non-reentrant shaped portion which extends into the housing chamber and an internal bore through which flow proceeds, the non-reentrant shaped portion of the extension being defined by an outer diameter, and

a filter assembly comprising a filter material and at least one end cap, the end cap including a substantially annularly shaped bore defined by a wall having an inner diameter, the inner diameter of the bore wall being smaller than the outer diameter of the non-reentrant shaped portion of the extension at ambient temperatures when the non-reentrant shaped portion is not disposed within the end cap bore,

the filter assembly being disposed within the housing chamber in the flow path, the non-reentrant shaped

5,510,026

13

portion being disposed within the end cap bore during use, the extension and the ends cap being joined to one another by thermal expansion and contraction such that there is an interference between the inner diameter and the outer diameter that induces stress in the end cap, the end cap bore and the non-reentrant shaped portion of the extension being sealed to one another under elastic stress at ambient temperatures so that the end cap bore fits tightly to the non-reentrant shaped portion without the presence of a sealing member.

34. The filter assembly arrangement of claim 33 wherein the substantially annularly shaped portion of the extension is of a non-reentrant shape.

35. The filter assembly arrangement of claim 34 comprising an annular projection along an outer annular surface of the extension.

36. The filter assembly arrangement of claim 35 wherein the end cap comprises an annular recess along the inner diameter of the annular end cap bore, the annular projection being disposed within the annular recess.

37. The filter assembly arrangement of claim 34 wherein the annular surface bevels outward to a substantially flat annular surface.

38. The filter assembly arrangement of claim 34 wherein the annular surface bevels outward to form a spherical section.

39. The filter assembly arrangement of claim 33 wherein the end cap comprises a polymeric material.

40. The filter assembly arrangement of claim 39 wherein the polymeric material comprises TEFLON® PFA.

41. The filter assembly arrangement of claim 19 wherein the substantially annularly shaped portion of the extension is metal.

42. The filter assembly arrangement of claim 39 wherein the substantially annularly shaped portion of the extension is stainless steel.

43. The filter assembly arrangement as claimed in claim 33 wherein the annularly shaped portion of the extension comprises a fitting coupled to one of the apertures.

44. The filter assembly arrangement as claimed in claim 43 wherein the end cap bore and the fitting are engaged by raising the temperature of the end cap to facilitate the placement of the end cap bore over the fitting and returning the temperature of the end cap to ambient temperature to provide a thermally contracted seal.

45. The filter assembly arrangement as claimed in claim 43 wherein the end cap bore and the fitting are engaged by lowering the temperature of the fitting to facilitate the placement of the end cap bore over the fitting and returning the temperature of the fitting to ambient temperature to provide a thermally expanded seal.

46. The filter assembly arrangement as claimed in claim 33 wherein the end cap bore and the substantially annularly shaped portion of the extension are engaged by raising the temperature of the end cap to facilitate the placement of the end cap bore over the substantially annularly shaped portion of the extension and returning the temperature of the end cap to ambient temperature to provide the seal.

47. The filter assembly arrangement as claimed in claim 33 wherein the end cap bore and the substantially annularly shaped portion of the extension are engaged by lowering the temperature of the substantially annularly shaped portion of the extension to facilitate the placement of the end cap bore over the substantially annularly shaped portion of the exten-

14

sion and returning the temperature of the substantially annularly shaped portion of the extension to ambient temperature to provide the seal.

48. The filter assembly arrangement of claim 33 wherein the outer diameter and the inner diameter are relatively dimensioned to provide maximum residual stress in at least one of the extension and the end cap for maximum shelf life of the filter arrangement.

49. The filter assembly arrangement of claim 33 wherein the seal between the end cap bore and the annularly shaped portion of the extension seals and secures the filter assembly to the housing assembly.

50. The filter assembly arrangement of claim 33 wherein the interference induces a stress in the end cap which is below the yield stress of the material from which the end cap is fabricated.

51. A filter arrangement comprising:

a housing assembly including a housing chamber having an aperture and a hollow fitting extending from the aperture into the housing chamber, the hollow fitting having an outer surface with a non-reentrant shape, and a filter assembly disposed in the housing chamber and including a filter material and an end cap connected to the filter material, the end cap having a bore with a wall, the wall being sealed tightly about the outer surface of the fitting by an interference fit at ambient temperatures, the end cap and the fitting being joined to one another by thermal expansion and contraction of at least one of the end cap and the fitting.

52. The filter arrangement of claim 51, wherein one of the bore wall and the non-reentrant shaped fitting comprises a polymeric material and the other of the bore wall and the non-reentrant fitting comprises a metallic material.

53. The filter arrangement of claim 52, wherein the bore of the end cap has a cylindrical shape along an axis of the bore where the bore wall is sealed to the fitting.

54. The filter arrangement of claim 53, wherein the non-reentrant shaped fitting extends outward to a substantially flat annular surface.

55. The filter arrangement of claim 54, wherein the filter assembly further includes a blind end cap mounted to the filter.

56. The filter arrangement of claim 51, wherein at least one of the end cap and hollow fitting is fabricated from a material that thermally expands and contracts.

57. The filter arrangement of claim 56, wherein the material includes a polymeric material.

58. The filter arrangement of claim 56, wherein the material includes a metallic material.

59. A filter arrangement as claimed in claim 51 wherein the bore wall has a recess which engages the outer surface of the fitting.

60. A filter arrangement as claimed in claim 51 wherein the bore wall has a constant shape along an axis of the bore where the bore wall is sealed to the fitting.

61. A filter arrangement as claimed in claim 51 wherein the outer surface of the fitting bulges outward from an axis of the fitting.

62. The filter arrangement of claim 51, wherein the non-reentrant shaped fitting has an outer diameter greater than the inner diameter of the bore of the end cap before the bore wall is sealed about the fitting.

* * * * *

RECYCLED

Exhibit P

US006174439B1

(12) United States Patent
Hopkins et al.

(10) Patent No.: US 6,174,439 B1
(45) Date of Patent: *Jan. 16, 2001

(54) FILTER PACKAGE

(75) Inventors: Scott D. Hopkins, Dryden; Daniel W. Spencer, Cortland; Joseph A. Perl, DeRuyter, all of NY (US)

(73) Assignee: Pall Corporation, East Hills, NY (US)

(*) Notice: Under 35 U.S.C. 154(b), the term of this patent shall be extended for 0 days.

This patent is subject to a terminal disclaimer.

(21) Appl. No.: 09/310,147

(22) Filed: May 12, 1999

Related U.S. Application Data

(63) Continuation of application No. 08/650,132, filed on May 8, 1996, now Pat. No. 5,928,516, which is a continuation-in-part of application No. 08/376,217, filed on Jan. 20, 1995, now abandoned, and a continuation-in-part of application No. PCT/US96/01348, filed on Jan. 19, 1996.

(51) Int. Cl.⁷ B01D 29/07

(52) U.S. Cl. 210/493.1; 210/636; 53/434; 53/469; 53/79; 53/167

(58) Field of Search 210/636, 257.2, 210/416.2, 232, 493.1; 422/21, 25, 26; 53/425, 469, 428, 440, 434, 79, 167; 206/205, 484

(56) References Cited**U.S. PATENT DOCUMENTS**

- | | | |
|-----------|--------|---------------------|
| 3,016,284 | 1/1962 | Trexler . |
| 3,088,255 | 5/1963 | Griem . |
| 3,229,813 | 1/1966 | Crowe, Jr. et al. . |

3,468,471	9/1969	Linder .
3,754,368	8/1973	Moore et al. .
4,176,070	11/1979	Sakurada et al. .
4,176,156	11/1979	Asanuma et al. .
4,177,620	* 12/1979	Daly et al. 53/425
4,295,563	10/1981	Becker et al. .
4,411,866	* 10/1983	Kanno 422/25
4,727,703	* 3/1988	Zahka 53/425
4,727,705	3/1988	Zahka .
4,810,469	* 3/1989	Masuhara 422/26
4,840,709	6/1989	Nejigaki et al. .
5,044,141	9/1991	Franchi .
5,164,166	11/1992	Stepanski et al. .
5,480,565	1/1996	Levin et al. .
5,556,591	* 9/1996	Jallerat et al. 210/321.89
5,928,516	* 7/1999	Hopkins et al. 210/636

FOREIGN PATENT DOCUMENTS

318667	2/1957	(CH) .
574356	4/1976	(CH) .
3724803	2/1989	(DE) .
0139822	5/1985	(EP) .
2784656 B2	9/1990	(JP) .

* cited by examiner

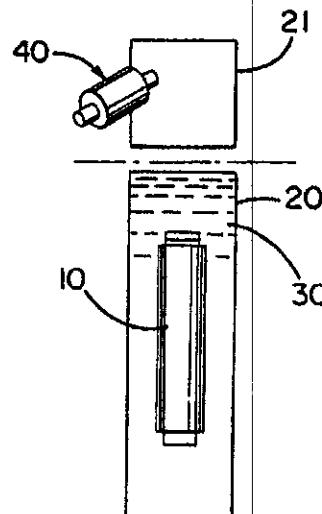
Primary Examiner—Ana Fortuna

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ABSTRACT

A method of forming a filter package includes disposing a filter and a liquid in a container with the filter immersed in the liquid and sanitizing the liquid and the filter while in the container. After sanitizing, the container may be hermetically sealed to enclose the sanitized liquid and filter. The container may be vented during sanitizing to allow vapor of the liquid to exit from the container and prevent pressure from building up in the container. The container may be either rigid or flexible.

23 Claims, 6 Drawing Sheets



U.S. Patent

Jan. 16, 2001

Sheet 1 of 6

US 6,174,439 B1

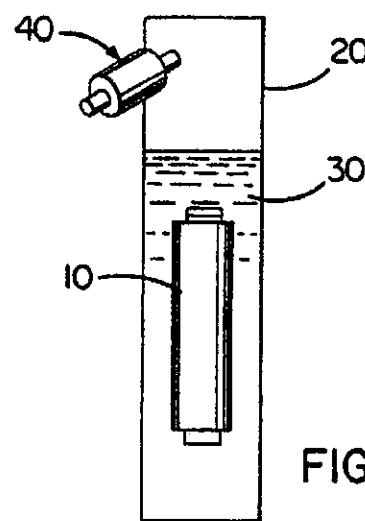


FIG. 1

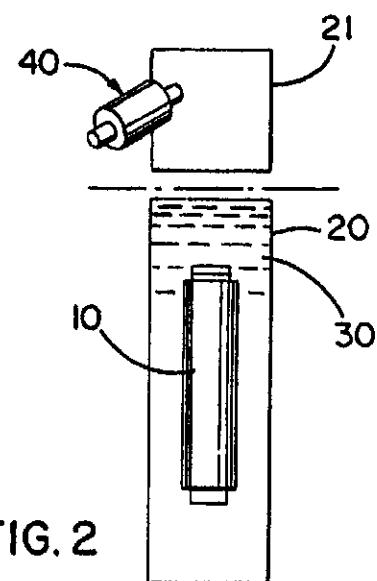


FIG. 2

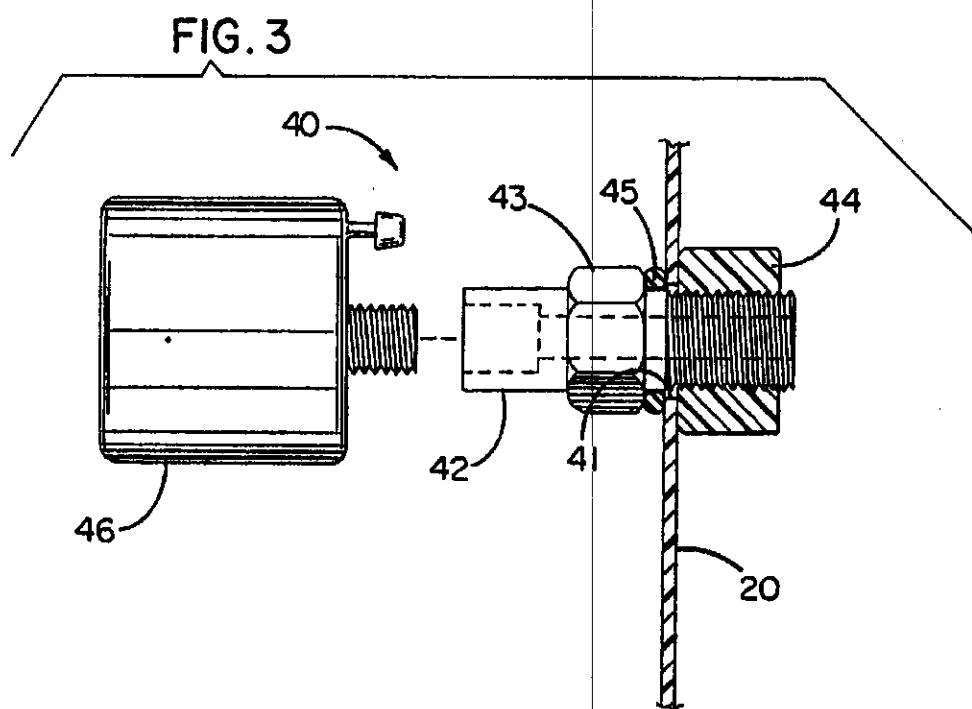


FIG. 3

U.S. Patent

Jan. 16, 2001

Sheet 2 of 6

US 6,174,439 B1

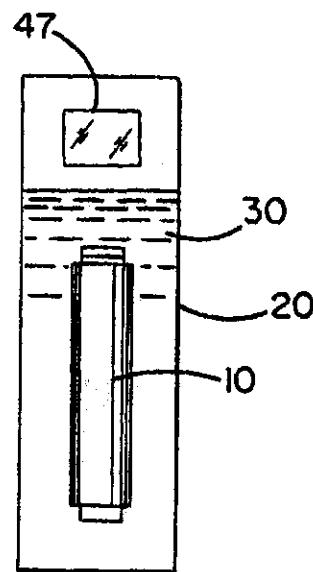
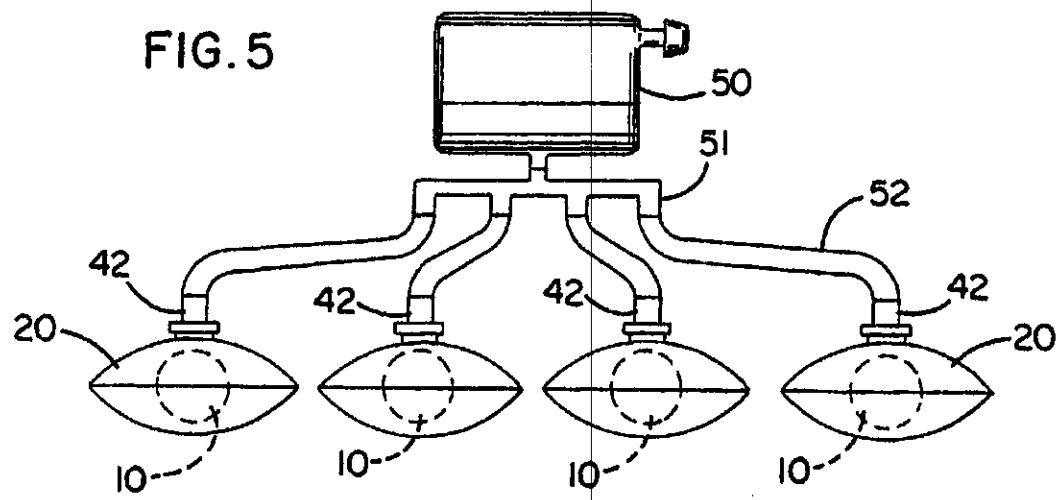


FIG. 4



U.S. Patent

Jan. 16, 2001

Sheet 3 of 6

US 6,174,439 B1

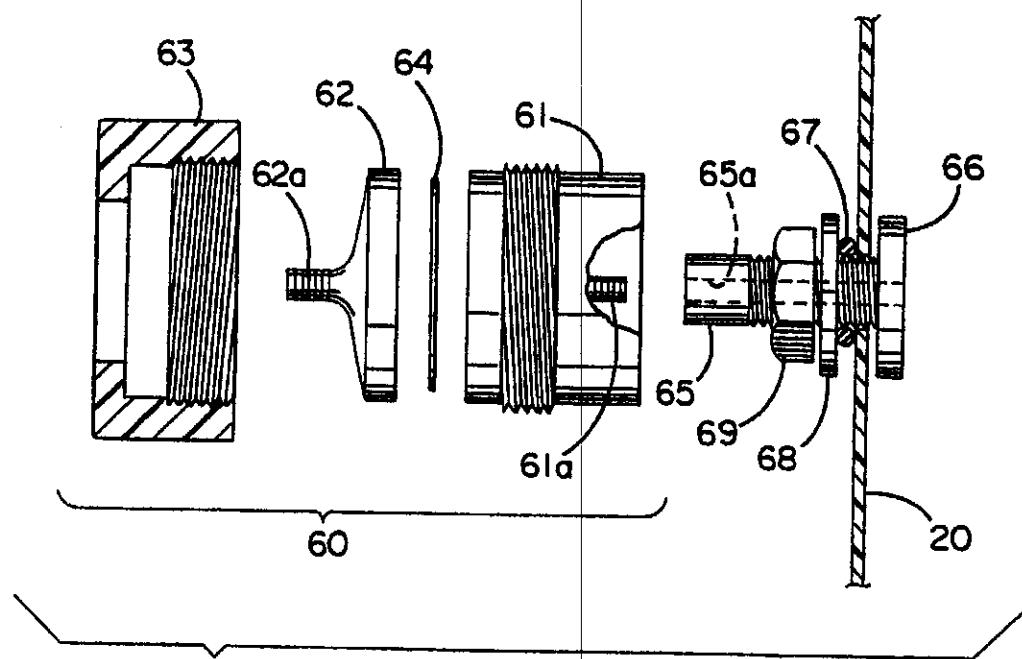


FIG. 6

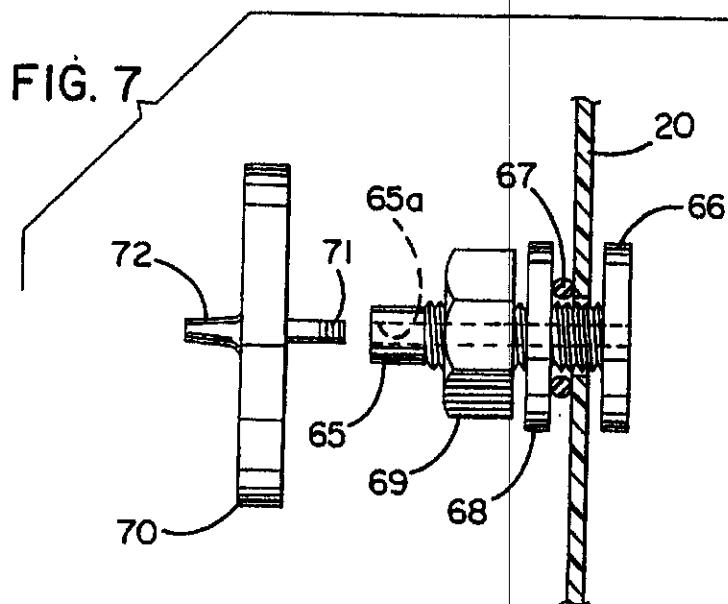


FIG. 7

U.S. Patent

Jan. 16, 2001

Sheet 4 of 6

US 6,174,439 B1

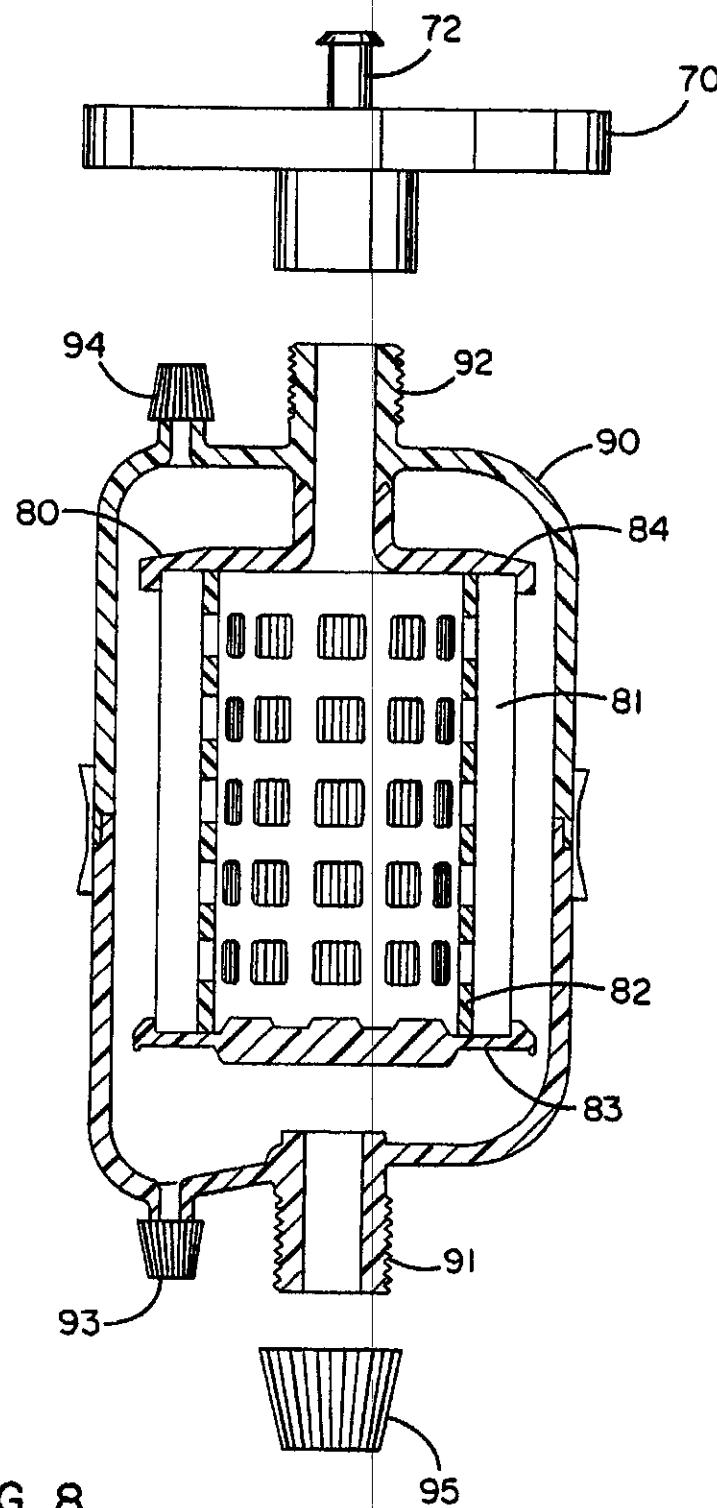


FIG. 8

U.S. Patent

Jan. 16, 2001

Sheet 5 of 6

US 6,174,439 B1

FIG. 9

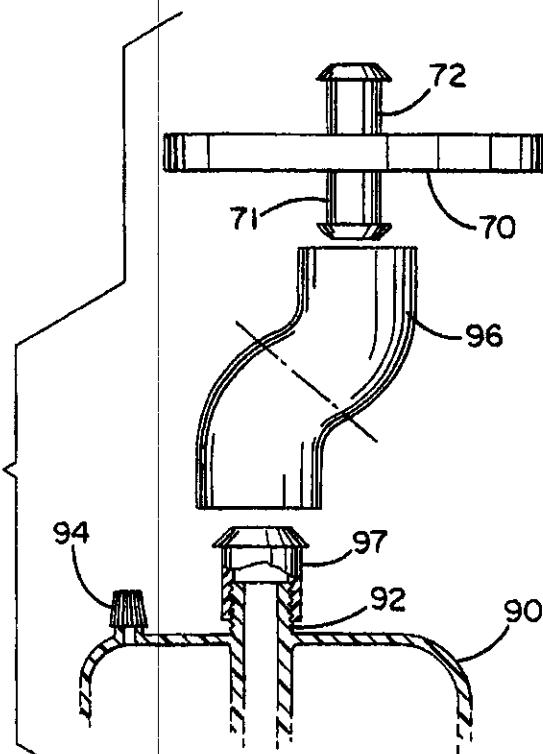
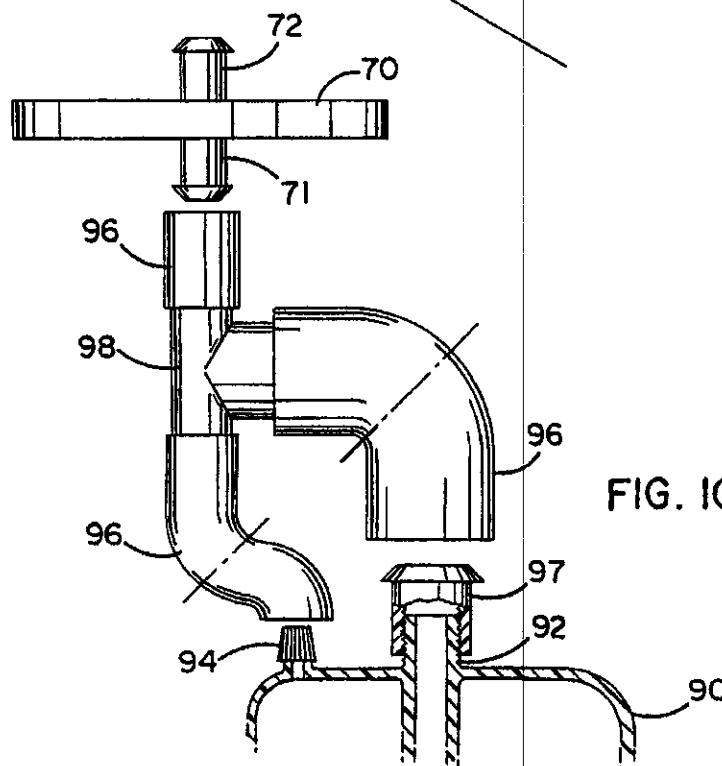


FIG. 10

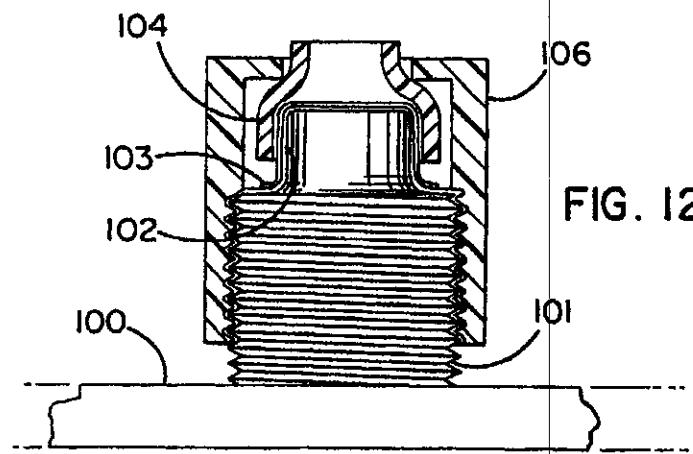
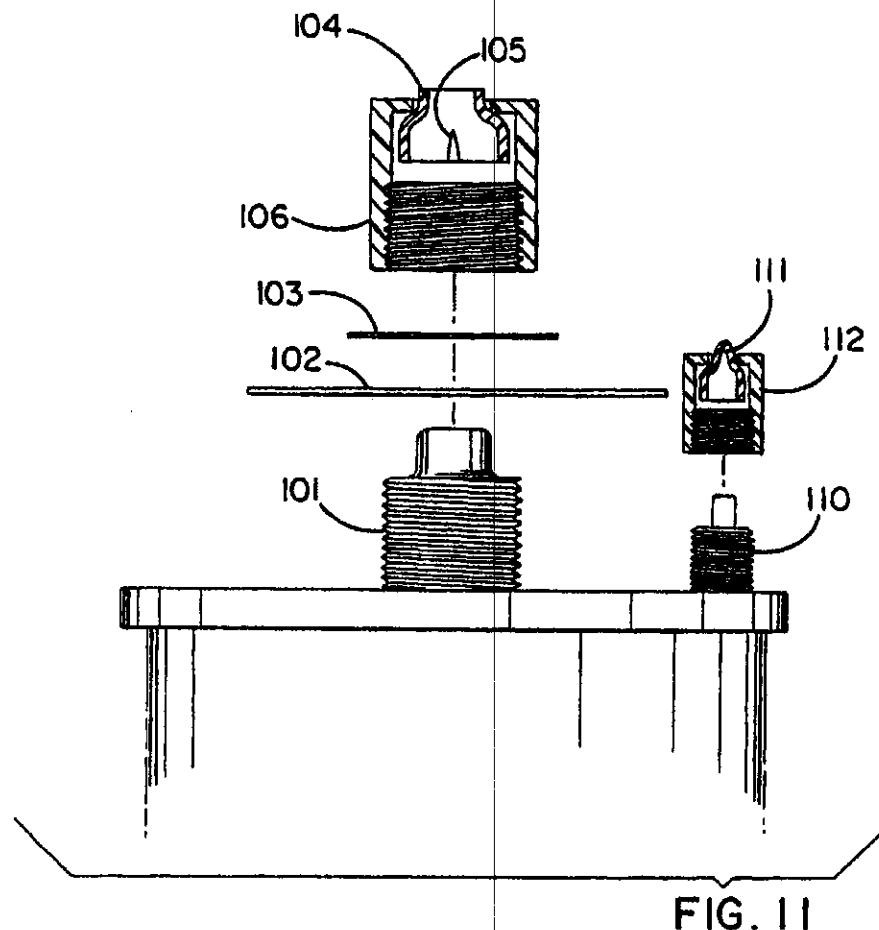


U.S. Patent

Jan. 16, 2001

Sheet 6 of 6

US 6,174,439 B1



US 6,174,439 B1

1

FILTER PACKAGE**REFERENCE TO RELATED APPLICATIONS**

This application is a continuation of U.S. patent application Ser. No. 08/650,132, filed on May 8, 1996, now U.S. Pat. No. 5,928,516, which is a continuation-in-part of U.S. patent application Ser. No. 08/376,217, filed on Jan. 20, 1995, now abandoned and of International Application No. PCT/US96/01348, filed on Jan. 19, 1996.

BACKGROUND OF THE INVENTION**1. Field of the Invention**

This invention relates to a filter package containing a filter immersed in a liquid and to a method of forming such a filter package. More particularly, it relates to a filter package the contents of which are sanitized and preferably sterilized.

2. Description of the Related Art

It is common for filters to be stored and shipped in a wet state, immersed in a suitable liquid within a package. There are several reasons for packaging a filter in this manner. Some filters are not readily wettable by the liquid which they are intended to filter and so are usually prewetted with another liquid having a lower surface tension to prepare the filter for filtration. As a service to the customer, some filter manufacturers perform prewetting at the factory where the filter is manufactured. In order to prevent the prewetted filter from drying out during storage or shipment, the filter is packaged in a sealed bag containing a suitable liquid which keeps the filter wetted until it is ready to be used.

Other types of filters, such as ultrafiltration and reverse osmosis membranes, are not "prewetted" by the manufacturer but are nevertheless shipped to the customer in a wet state in order to maintain their permselective properties. These filters are typically stored and shipped in packages containing a humectant such as glycerin which keeps the filter wet.

Another reason for packaging a filter in a wet state is that it is easier to ensure the cleanliness of such a filter than if it is packaged in a dry state. Thus, even filters which do not require prewetting and which do not need to be kept wet to maintain their filtering properties may be packaged in a wet state for reasons of cleanliness.

In order to give a filter package containing a wet filter a suitable shelf-life, hydrogen peroxide or other bactericide is usually added to the liquid within the package in order to prevent bacterial growth between the time of manufacture and the time that the purchaser opens the package.

Even though the amount of the bactericide is relatively small (typically around 3% in the case of hydrogen peroxide), in some applications, and particularly in the manufacture of semiconductors, the bactericide is an undesirable contaminant. Accordingly, there is a need for a filter package containing a filter in a wet state which has a long shelf-life yet which contains substantially no contaminants.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide a filter package containing a filter and a noncontaminating liquid with substantially no contaminants, the filter package having a long shelf life without employing bactericides.

It is another object of the present invention to provide a method of preparing such a filter package.

In accordance with one aspect of the invention, a filter package which enables the filter to be stored and shipped in

2

a wet state without the filter drying out comprises a sanitized filter cartridge, a flexible bag which surrounds the filter cartridge, and sanitized water hermetically sealed inside the bag. The filter is capable of removing particulates from a fluid and has a porous filter medium including a plurality of pores through which the first liquid can pass between upstream and downstream sides of the filter during filtration to remove particulates from the fluid. The flexible bag has walls extending between an upper end and a lower end of the bag, and the walls comprise a polymeric material impervious to microorganisms and liquid water. The sanitized water substantially permeates the pores of the filter medium and immerses substantially 100% of a volume of the filter cartridge. The bag is hermetically sealed around the filter cartridge with substantially all air excluded from the bag.

In accordance with another aspect of the invention, a filter package which has a long shelf life and contains substantially no contaminants includes a sanitized filter cartridge, a flexible bag hermetically sealed around the filter cartridge, and sanitized water hermetically sealed inside the bag. The filter is capable of removing particulates from a fluid and has a porous filter medium including a plurality of pores through which the fluid can pass between an upstream side and a downstream side of the filter as particulates are removed from the fluid. The flexible bag has an inside surface, an outside surface, and walls comprising a material impervious to microorganisms and liquid water. The sanitized water contacts the inside surface of the bag, substantially permeates the pores of the filter medium so that the filter will not dry out during storage and shipment, and immerses substantially 100% of the volume of the filter cartridge. Substantially all air is excluded from the bag.

In accordance with a further aspect of the invention, a filter package comprises a flexible bag, a sanitized filter cartridge disposed in the bag, and sanitized water hermetically sealed inside the bag. The flexible bag has an inside surface, an outside surface, and walls comprising a material impervious to microorganisms and liquid water. The sanitized filter includes a filter medium having pores through which a fluid can pass between an upstream side and a downstream side of the filter to be filtered by the filter medium. The sanitized water contacts the inside surface of the bag, substantially permeates the pores of the filter medium, and immerses substantially 100% of a volume of the filter cartridge.

The sanitized water within the bag is preferably one which contains substantially no substances, such as bactericides or other organic or inorganic substances, which could be considered contaminants with respect to the filter or its intended use. For example, the level of contaminants in the sanitized water is preferably at most in the parts per billion range. In preferred embodiments, the sanitized water comprises deionized water having an initial resistivity of at least 18 MΩ·cm.

The bag preferably comprises a thin-walled, flexible bag such as a bag formed from a polymeric material. The walls of the bag may be transparent or translucent.

A filter package according to the present invention is not restricted to one having any particular type of filter. For example, the filter may be either hydrophilic or hydrophobic, it may be a filter for filtration of gases, liquids, slurries, or mixtures of more than one phase, and the mechanism by which it performs filtration is not important. The filter may preferably be a filter cartridge and may include a pleated filter element. A few examples of various types of filters which may be employed in the present

invention are particulate filters, particularly for use in the semiconductor industry, coalescers, ultrafiltration membranes, and reverse osmosis membranes.

The present invention also provides a method of preparing a filter package including disposing a filter and a liquid in a container with the filter immersed in the liquid. The liquid and the filter are then sanitized and preferably sterilized while in the container. If desired, the container may be hermetically sealed after sanitizing.

The contents of the filter package of the present invention are at least sanitized, i.e., all or substantially all non-spore producing microorganisms are killed, and preferably the contents of the package are fully sterilized. In this description, "sterilizing" is included within the scope of the term "sanitizing". Thus, a sanitized filter package according to the present invention may be one which has been fully sterilized or one which has been sanitized without being fully sterilized.

The sanitizing can be performed in any manner which will not damage or degrade the filter or the container. In preferred embodiments, sanitizing is performed by heating the liquid and the filter within the container. When sanitizing is performed by heating, the container may be vented during sanitizing to permit vapor of the liquid to exit from the container and prevent the build-up of pressures which could damage the container.

If desired, the filter may be prewetted prior to being immersed in liquid in the container so that it can be completely wetted by the liquid in which it is immersed.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic elevation of a filter package according to the present invention prior to sanitizing.

FIG. 2 is a schematic elevation of the filter package of FIG. 1 after being hermetically sealed.

FIG. 3 is a partly cross-sectional view of the venting mechanism of the embodiment of FIG. 1.

FIG. 4 is a schematic elevation of a filter package in which a venting mechanism is formed by a semipermeable membrane.

FIG. 5 is a schematic plan view of an assembly including a plurality of filter packages connected to a common vent filter.

FIGS. 6 and 7 are side elevations of different types of vent filters which can be employed in the present invention.

FIG. 8 is a cross-sectional elevation of a filter assembly which can be formed into a filter package according to the present invention.

FIG. 9 is a partially cross-sectional view illustrating the use of flexible tubing to connect a filter assembly to a vent filter.

FIG. 10 is a partially cross-sectional view illustrating a method of venting a filter housing on both the upstream and downstream sides of a filter element.

FIG. 11 is a partially cross-sectional exploded view of a portion of a filter assembly having a filter membrane mounted directly on a fluid port of the filter assembly.

FIG. 12 is a partially cross-sectional view of the outlet of the filter assembly of FIG. 11 as it appears during sanitizing.

DESCRIPTION OF PREFERRED EMBODIMENTS

FIGS. 1 and 2 schematically illustrate a method of preparing a filter package according to the present invention. A

filter 10 and a noncontaminating liquid 30 are placed in a container, such as a bag 20, to immerse the filter 10 in the noncontaminating liquid 30. After the bag 20 is closed to prevent liquid or microorganisms from entering it, the filter 10 and the noncontaminating liquid 30 are sanitized and preferably sterilized while in the bag 20. Next, as shown in FIG. 2, the bag 20 is preferably hermetically sealed to obtain a completed filter package.

Prior to being placed into the bag 20, the filter 10 may be prewetted so that it can be readily wetted by the fluid with which it is to be used and thus be ready for use by the customer. Alternatively, depending on the nature of the filter 10, its end use, and the requirements of the purchaser, the filter 10 may be packaged without being prewetted. However, if a high degree of cleanliness of the filter 10 is important and if the filter 10 is not readily wettable in a dry state by the noncontaminating liquid 30, then it is preferable to prewet the filter 10 such that the noncontaminating liquid 30 can readily penetrate the pores of the filter 10.

The filter 10 can be prewetted using any known method appropriate for the type of the filter 10. For example, the standard prewetting procedures recommended by the manufacturer of the filter 10 are suitable. A common method of prewetting is to immerse the entire filter 10 in a vessel containing a prewetting liquid having a low surface tension, such as isopropyl alcohol or methyl alcohol, and to allow the prewetting liquid to permeate the filter medium. The prewetting liquid is preferably filtered prior to use in order to remove any possible particulate contaminants from the prewetting liquid. If the prewetting liquid would be a contaminant in the fluid system in which the filter 10 is to be used, the prewetting liquid is preferably flushed out of the filter 10 using a suitable noncontaminating liquid, such as deionized water. Flushing of the filter 10, with a noncontaminating liquid can be performed using conventional procedures. After prewetting and possibly flushing, the filter 10 is disposed in the bag 20 before the filter 10 has had a chance to dry.

The bag 20 or other container in which the filter 10 is packaged is not restricted to any particular type and can be either rigid or flexible. It can be any size and shape which enables it to completely enclose the filter 10 and the noncontaminating liquid 30 in which the filter 10 is immersed. If the filter 10 is durable enough to withstand forces likely to be encountered during storage and shipment, a flexible, thin-walled bag 20 is particularly suitable as the container since the bag 20 can be inexpensively manufactured and is easy to seal and handle, impermeable to the noncontaminating liquid 30 and to microbes and is capable of withstanding the conditions occurring during sanitizing without decomposing or releasing contaminants into the noncontaminating liquid 30. The bag 20 is also preferably impermeable to vapor of the noncontaminating liquid 30 and other gases, and to any liquids which the bag 20 is likely to contact during storage or shipment. High-temperature thermoplastic fluoropolymers are particularly suitable for use as the bag material because they are strong, lightweight, readily sealed, and can withstand sterilizing temperatures. Examples of suitable materials for the bag 20 when the noncontaminating liquid 30 is water are PFA (perfluoroalkoxy), FEP (fluorinated ethylene propylene), PVDF (polyvinylidene fluoride), and ECTFE (ethylene chlorotrifluoroethylene). Non-polymeric materials such as metal foils may also be used, as may a combination of one or more materials, such as a laminate of aluminum foil and Mylar film.

If sanitizing is performed using heating, nuclear irradiation, ozone, or ultrasonics, for example, the bag 20

need not be permeable to light. However, it may be easier to seal the bag 20 if it is made of a transparent or translucent material so that the filter 10 and the level of the noncontaminating liquid 30 are visible to the person performing the sealing.

The type of noncontaminating liquid 30 placed into the bag 20 and its purity can be selected in accordance with the characteristics of the filter 10 and the fluid system in which the filter 10 is to be employed. A preferred noncontaminating liquid is ultrapure deionized water having an initial resistivity of at least 18 MΩ-cm and more preferably at least 18.1 MΩ-cm. The initial resistivity of the deionized water refers to its resistivity prior to use and at the time it is placed into the bag 20. Due to the presence of substances in the air, the filter 10, or the inside of the bag 20 which may come into contact with the deionized water during assembly of the filter package, the resistivity of the deionized water may decrease somewhat from its initial resistivity after it is placed into the bag 20. However, the level of contaminants in the deionized water within the bag 20 during sanitizing is preferably at most in the parts per billion range. Thus, during sanitizing, the bag 20 preferably contains essentially only the filter 10, the noncontaminating liquid 30, and possibly air or other gas above the surface of the noncontaminating liquid 30. No bactericides are present in the bag 20. When the noncontaminating liquid 30 is introduced into the bag 20, the filter 10 and the bag 20 may be disposed in an atmosphere of a gas having low solubility in the noncontaminating liquid 30 to prevent gases in the air from being dissolved in the liquid 30. For example, when the noncontaminating liquid 30 is deionized water, the liquid 30 may be introduced into the bag 20 inside a nitrogen atmosphere to prevent CO₂ in the air from dissolving in the liquid 30. However, in general, gases present in ordinary clean atmospheric air are not contaminants with respect to the filter 10, so it is typically not necessary to prevent them from contacting the noncontaminating liquid 30.

The filter 10 can be of any type and shape capable of being sanitized. For example, it may have a pleated or nonpleated filter medium and may include conventional equipment such as a perforated core, an outer cage, one or more end caps, and sealing members (O-rings, etc.) for connecting the filter 10 to a fluid system. The filter 10 may be in the form of a cartridge intended for installation in a housing. Alternatively, it may already be installed in a housing, as long as the housing does not interfere with prewetting and sanitizing. For example, the filter 10 can be installed in a housing having an opening through which a prewetting liquid and then the noncontaminating liquid 30 can be introduced to thoroughly contact the filter 10.

If the filter 10 is to be used within a short length of time after being packaged, such as on the same day, it may be sufficient to subject the contents of the bag 20 to a high degree of sanitization rather than to sterilization. However, in order to give the filter package as long a shelf life as possible, it is preferable to subject the entire contents of the bag 20, including the filter 10 and the noncontaminating liquid 30, to sterilization.

Any known method of sanitizing which will not introduce contamination into the bag 20 or damage the filter 10 or the bag 20 can be used, such as sanitizing using nuclear irradiation, ultraviolet light, ozone, heat, or ultrasonics. Sterilization by heating of the noncontaminating liquid 30 to a sterilizing temperature is preferred because it is simple, reliable, and inexpensive. Heating can be performed in a variety of ways, such as by disposing the bag 20 in an autoclave, in a microwave oven, in a pressure cooker, or in

a vessel of boiling water or other liquid at a sterilizing temperature. During sanitizing, the filter 10 is preferably immersed in the noncontaminating liquid 30 in the bag 20 both before and after sanitizing to prevent pores of the filter 10 from drying out during the sanitizing process. More preferably, it is mostly immersed (at least 50% of its volume), and most preferably it is entirely immersed in the noncontaminating liquid 30. If the filter 10 is negatively buoyant in the noncontaminating liquid 30, the filter 10 may be completely immersed simply by filling the bag 20 with a sufficient amount of the noncontaminating liquid 30. If the filter 10 floats in the noncontaminating liquid 30, it may be desirable to hold the filter 10 beneath the surface of the noncontaminating liquid 30 so as to completely immerse the filter 10, such as by pinching the bag 20 from the outside using a clamp disposed below the surface of the noncontaminating liquid 30 and above the top of the filter 10 to prevent the filter 10 from floating to the surface. During sanitizing, care is preferably taken that the bag 20 does not come into contact with any members which are at a temperature which could produce thermal deformation of the bag 20 or the filter 10. Care should also be taken not to boil the inside of the bag 20 dry. The sanitizing conditions, such as the heating temperature and the length of time for which heating is carried out, can be standard conditions. An example of suitable, conventional sterilizing conditions in an autoclave are 1 hour at a gauge pressure of 15 psi and a temperature of approximately 120° C. To reduce the risk of contamination, it may be desirable to perform the sanitizing in a clean room.

If the filter 10 is of a type having a blind end cap and an open end cap, the filter 10 is preferably placed in the bag 20 with the open end cap higher than the blind end cap so that air can escape from the center of the filter 10 through the open end cap and be displaced by the noncontaminating liquid 30.

In some cases, the heating of the filter 10 during sanitizing may produce leaching of extractables from the filter 10 into the noncontaminating liquid 30. In order to reduce the amount of leaching, the filter 10 may be pretreated prior to insertion into the bag 20 by immersion in hot deionized water (preferably at approximately 160 to approximately 200° F., such as at 165° F.=approximately 74° C.) to leach out extractables prior to sanitizing.

The upper end of the bag 20 is preferably closed during and after sanitizing in a manner such that contaminants cannot enter the bag 20. Closure of the upper end can be performed in any suitable manner which does not introduce contamination, such as by heat sealing. However, even though the bag 20 is preferably closed, it is preferably not hermetically sealed as a whole during sanitizing but rather is closed in a manner such that vapor of the noncontaminating liquid 30 and air can exit from the bag 20 while dust, microorganisms, and other contaminants are prevented from entering. When sanitizing takes place by heating, the pressure in the bag 20 will increase due to an increase in the vapor pressure of the noncontaminating liquid 30, boiling of the noncontaminating liquid 30, and/or gases in the noncontaminating liquid 30 coming out of solution. If the bag 20 is hermetically sealed during sanitizing, it is desirable to take steps to ensure that the pressure which builds up within the bag does not rupture or otherwise damage the bag 20, such as making the walls of the bag 20 sufficiently thick to resist the internal pressure without damage, or pressurizing the inside of the autoclave with air to reduce the amount of swelling of the bag 20 during heating. However, increasing the wall thickness of the bag 20 raises costs and makes the

bag 20 more difficult to handle, while pressurizing the autoclave reduces the efficiency of heating in the autoclave. Therefore, a preferred method of preventing damage to the bag 20 by an increase in internal pressure is to provide the bag 20 with a venting mechanism 40 which is able to release vapor of the noncontaminating liquid 30 and other gases generated during heating which could cause deformation or rupture of the bag 20.

A venting mechanism 40 can be installed on the bag 20 in any location in which it can allow vapor of the noncontaminating liquid 30 or other gases to escape from the bag 20. During sanitizing by heating, the bag 20 is preferably positioned so that the venting mechanism 40 is in an upper portion of the bag 20 where air and other gases can accumulate. The venting mechanism 40 can be structured in any manner which allows the discharge of vapor of the noncontaminating liquid 30 and other gases from the bag 20 during sanitizing. FIG. 3 illustrates an example of a venting mechanism 40 in detail. It includes a vent hole 41 formed in a wall of the bag 20 and a hollow vent tube 42 passing through the vent hole 41 and communicating between the inside and outside of the bag 20. The vent tube 42 may be omitted, but it provides a convenient way of connecting the vent hole 41 to external hardware. The vent tube 42 is secured to the bag 20 by means of a nut 44 disposed inside the bag 20 which screws onto external threads formed on the inner end of the vent tube 42. A nut 43 is integrally formed on the outer end of the vent tube 42 on the outside of the bag 20. In order to form a hermetic seal around the vent hole 41, a seal member such as an elastomeric O-ring 45 is disposed around the vent tube 42 near the periphery of the vent hole 41 between the wall of the bag 20 and one of the nuts 43 and 44. The O-ring 45 is pressed into sealing contact with the bag 20 by tightening of the nuts 43 and 44. The O-ring 45 may be disposed on either the inside or the outside of the bag 20, but as the O-ring 45 may possibly introduce contaminants, it is preferably on the outside of the bag 20.

The vent tube 42 and the nuts 43 and 44 can be made of any corrosion resistant material which can resist the temperatures occurring during sanitizing. Examples of suitable materials are polymers such as FEP, PFA, PVDF, and ECTFE and metals such as stainless steel.

A wide variety of other methods can be employed to scalably mount the vent tube 42 on the bag 20, such as the use of bulkhead fittings. Furthermore, the vent tube 42 may be permanently connected to the bag 20 by a method such as welding. However, it is often advantageous if the vent tube 42 is detachable from the bag 20 so that the vent tube 42 can be reused with different bags.

Another possible type of venting mechanism is a sheet of a semipermeable membrane which is permeable to water vapor but impermeable to liquid water and microorganisms, such as a PTFE (polytetrafluoroethylene) membrane forming a section of the bag 20. FIG. 4 illustrates an embodiment in which a venting mechanism comprising a semipermeable membrane 47 of PTFE forms a section of the wall of the bag 20, the remainder of the bag 20 being made of PFA. The membrane 47 is located in an upper portion of the bag 20, in substantially the same location as the venting mechanism 40 of FIG. 1. Sanitizing is performed using this bag 20 in the same manner as with the bag 20 illustrated in FIG. 1.

Alternatively, the entire bag 20 can be made of a semi-permeable membrane, such as a PTFE membrane, which is permeable to water vapor but not to liquid water or microbes, in which case a separate venting mechanism becomes unnecessary. However, a bag 20 made of a material

which is permeable to water vapor is less preferred, since water vapor can pass through the bag 20 during storage and condense on the outside of the bag 20, making the bag 20 awkward to handle. In addition, over time, all of the non-contaminating liquid may pervaporate from the bag 20, leaving the filter 10 dried out.

Because of the provision of the venting mechanism 40, very little internal pressure acts on the walls of the bag 20 during sanitizing, so the walls of the bag 20 can be quite thin. For example, a bag made of PFA with a wall thickness of 0.002-0.030 inches, such as 0.005 inches has been found to work quite well for sterilization in an autoclave at 15 psi gauge. Decreasing the wall thickness of the bag 20 is advantageous because it decreases material costs and makes the bag 20 easier to seal.

In order to prevent microorganisms and other contaminants from entering the bag 20 through the vent hole 41, either during or after sanitizing, a vent filter 46 which is able to prevent the passage of bacteria or other microorganisms therethrough is preferably hermetically connected to the vent tube 42 so that all air entering the vent tube 42 from outside the bag 20 must pass through the vent filter 46. The term vent filter here refers to any type of filter which allows the passage of vapor of the noncontaminating liquid, and the vent filter need not be a filter intended exclusively for use in venting. Preferably, the vent filter 46 allows the passage of air. An example of a suitable vent filter 46 is a sterilizing grade filter for air filtration. A sterilizing grade filter or filter medium is typically defined as one having a removal rating of 0.2 μm . Depending on the environment in which the bag 20 is disposed following sanitizing, a vent filter may be unnecessary, or one having a different removal rating, i.e., a non-sterilizing grade filter may be employed. When the noncontaminating liquid 30 in the bag 20 is water, the vent filter 46 is preferably hydrophobic, i.e., having a critical wetting surface tension of less than approximately 50 dynes/cm, so that it does not become wetted during sanitizing, since wetting could prevent the flow of gases through the vent filter 46. In addition, if the noncontaminating liquid 30 is water, a hydrophobic vent filter 46 prevents the noncontaminating liquid 30 from leaking out of the bag 20, even when the bag 20 is turned upside down, making it easier to store the bag 20. However, if the vent filter 46 can be prevented from wetting during sanitizing, a hydrophilic vent filter can also be employed. The vent filter 46 may have any shape and may be either pleated or nonpleated. An example of a suitable vent filter is a DFA4001FRP filter assembly available from Pall Corporation. This filter has a PTFE dual-layer filter medium, an internal core, and caps made of polypropylene, and a critical wetting surface tension of less than 30 dynes/cm. Such a filter, when not wetted, is impermeable to liquid water but is permeable to liquids having a surface tension smaller than 30 dynes/cm. The vent filter 46 can be installed in any manner providing a seal which prevents microorganisms from bypassing the vent filter 46, and it may be either permanently or detachably connected to the bag 20. However, a detachable connection is preferred to permit the vent filter 46 to be reused. For example, as shown in FIG. 3, the vent filter 46 and the vent tube 42 can be connected by a threaded coupling.

The noncontaminating liquid 30 can be introduced into the bag 20 in any desired manner. For example, it can be introduced through the open end of the bag 20 before it is closed, or it can be introduced through the vent tube 42 of the venting mechanism after the bag 20 has been closed and before the vent filter 46 has been installed on the vent tube 42.

After the contents of the bag 20 have been sanitized, the bag 20 is preferably hermetically sealed. Since a hydrophobic vent filter can prevent leakage from the bag 20 as well as prevent water and microbes from entering the bag 20, it is not mandatory to hermetically seal the bag 20, but doing so allows the venting mechanism 40 to be detached from the bag 20 and makes the bag 20 easier to handle. Before sealing is performed, it may be desirable to allow the bag 20 to cool to a comfortable handling temperature. During cooling, the vent filter 46 prevents microbes and other contaminants bag from entering the bag 20 and maintains the contents of the bag 20 sterile. Any known method of hermetically sealing the bag 20 can be employed. When the bag 20 is made of a polymeric material, heat sealing is particularly suitable. Other methods such as ultrasonic sealing and vibration welding can also be employed. The bag 20 can be sealed at any desired location, including below the surface of the noncontaminating liquid 30 so as to exclude all air from the inside of the bag 20. While preferably the bag 20 contains no air above the surface of the noncontaminating liquid 30 after being sealed, since any air in the bag 20 has been sterilized and is at 100% relative humidity, it is not detrimental to have some air remaining in the bag 20 after sealing because the air will neither contaminate nor dry out the filter 10. After the bag 20 is sealed, the upper portion of the bag 20 including the venting mechanism 40 can be detached from the lower portion of the bag 20 and salvaged for reuse. If the venting mechanism 40 does not need to be reused, it can be left attached to the bag 20, but in this case it is preferably disabled from venting, since water vapor passing through the venting mechanism 40 could condense on the outer surface of the bag 20 during storage and form a puddle of water surrounding the bag 20. In the embodiment of FIG. 1, the venting mechanism 40 could be disabled by forming a seal around the vent hole 41, such as by heat sealing, to isolate the vent hole 41 from the inside of the bag 20.

It may be desirable to simultaneously sanitize a plurality of filters 10 housed in individual bags 20 or other containers. Instead of equipping each of a plurality of bags 20 with its own vent filter, the vent tubes 42 of the plurality of bags 20 can be connected to a single vent filter 50 by a manifold 51 and hoses 52, as schematically illustrated in FIG. 5. The vent filter 50 is selected to be large enough to provide filtration of air for all of the bags 20. The entire assembly of the plurality of bags 20 and the vent filter 50 can be placed in an autoclave at one time to sanitize the filters 10 as a batch.

Alternatively, a plurality of filters 10 can be disposed in a single bag 20 like that shown in FIG. 1 so as to simultaneously sanitize the plurality of filters 10.

FIGS. 6 and 7 illustrate other examples of vent filters through which the bag 20 can be vented during sanitizing. The vent filter 60 of FIG. 6 comprises a commercially available filter holder and a sheet of a filter medium 64 disposed inside the filter holder. The filter holder has a generally cylindrical housing including a base 61 and a cover 62 between which the filter medium 64 can be placed. The base 61 and the cover 62 are sealed to each other by a nut 63 which surrounds the cover 62 and screws onto external threads formed on the base 61. One or both of the base 61 and the cover 62 may include a perforated support plate for supporting the filter medium 64. The filter holder is usually purchased without the filter medium 64, which is installed by the user. First and second fluid ports 61a and 62a communicating with opposite sides of the filter medium 64 when the filter holder is assembled extend from the base 61 and the cover 62, respectively. The base 61 is partly cut away in the figure to show the first fluid port 61a. Filter holders of

this and other types which enable a filter medium to be installed and replaced by the user are available from a variety of sources, such as Cole-Parmer Instrument Company of Niles, Ill. The filter medium 64 which is supported by the filter holder can be one having any desired properties. An example of a suitable filter medium 64 for use in the present invention is a hydrophobic, sterilizing grade membrane filter medium of PTFE.

The bag 20 in this embodiment is equipped with a hollow vent tube 65 having a central bore 65a extending through its length. A hollow circular flange 66 having an outer diameter larger than that of the vent tube 65 is formed on the inner end of the vent tube 65. The vent tube 65 extends through a hole in the wall of the bag 20, with the flange 66 disposed on the inside of the bag 20. A sealing member such as an O-ring 67, a washer 68, and a nut 69 are mounted on the vent tube 65 on the outside of the bag 20. The nut 69 is threadingly engaged with external threads formed on the vent tube 65. When the nut 69 is tightened the washer 68 is urged towards the flange 66, and as a result, the bag 20 is compressed between the O-ring 67 and the flange 66, causing the O-ring 67 to be pressed into sealing contact with the bag 20 to form a seal around the hole in the bag 20. The O-ring 67 may be separate from the washer 68, or it may be attached to the washer 68 by an adhesive, for example. The vent tube 65 may be fluidly connected to either of the fluid ports 61a and 62a of the filter holder in any suitable manner. For example, the inner bore 65a of the vent tube 65a may be formed with internal threads which mate with external threads formed on the fluid ports. Alternatively, the vent tube 65 and one of the fluid ports of the vent filter 60 can be connected by a hollow connecting member such as a pipe or flexible tubing.

A vent filter comprising a filter holder which can be assembled and disassembled by the user has a number of useful attributes. Filter holders are available in a variety of sizes, so the user can select a filter holder capable of supporting a filter medium having a surface area appropriate for the application. Since the filter medium can be readily installed in the filter holder by the user, the filter medium can be discarded and replaced when necessary while the filter holder can be reused, making the filter holder economical to employ. In addition, the user has great freedom of choosing a filter medium for use with the filter holder.

The vent filter 70 shown in FIG. 7 comprises a commercially available, disposable filter unit referred to as a syringe filter because it is adapted for mounting on a medical syringe. It includes an unillustrated filter medium sealed inside a plastic housing having first and second fluid ports 71 and 72 communicating with opposite sides of the filter medium. Syringe filters are available with a variety of different filter media. An example of a suitable filter medium for a syringe filter for use in venting a filter package according to the present invention is a hydrophobic, sterilizing grade membrane filter medium. A syringe filter will usually include, within its housing, a perforated support plate on one or both sides of the filter medium. The vent filter 70 can be connected to the bag 20 in any suitable manner, such as by a vent tube 65 like that shown in FIG. 6. Syringe filters are available with a variety of fittings, and the structure of the vent tube 65 and the type of vent filter 70 may be selected so that the two can be connected directly to each other. In FIG. 7, the first fluid port 71 of the vent filter 70 is equipped with external threads which can be screwed into internal threads formed in the outer end of the vent tube 65. Alternatively, the vent filter 70 may be indirectly connected to the vent tube 65 by a flexible hose or a connecting pipe, for example.

US 6,174,439 B1

11

At the completion of sanitizing, the vent filters 60 and 70 may be left attached to the bag 20, or they may be detached after the bag 20 has been sealed, in the manner shown in FIG. 2.

If the venting mechanism comprises a semipermeable membrane, as in the embodiment of FIG. 4, and if the membrane 47 is impermeable to microbes, a vent filter is unnecessary.

When sanitizing is performed by heating the filter in a chamber such as an autoclave or an oven, the venting mechanism may vent to either the inside or the outside of the chamber. It is generally simpler if venting is performed to the inside of the chamber, i.e., if the venting mechanism is disposed inside the chamber with the filter. In this case, the venting mechanism is preferably made of materials which can withstand the conditions within the chamber during sanitizing.

As stated above, the container of a filter package according to the present invention may be a rigid container. Sanitizing of a filter in a rigid container, such as a housing for the filter, can be performed in much the same way as sanitizing of a filter in a flexible container, such as a flexible bag. A rigid container refers to one which maintains a substantially constant shape and dimensions without being supported, in contrast to a flexible container such as a flexible bag which is readily deformed and may collapse under its own weight if not internally or externally supported. A rigid container of a filter package according to the present invention may be made of any desired material, such as a metal or a polymeric material. A filter to be sanitized in a rigid container may be prewetted prior to sanitizing, and it may be pretreated in hot deionized water to leach out extractables. Prewetting and pretreatment may be performed either before or after the filter is installed in the container. However, when the container is a filter housing, it is usually easier to perform prewetting and pretreatment after the filter has been installed in the housing to form a filter assembly. Prewetting and pretreatment can be performed by immersing the filter assembly in a suitable liquid or by passing the liquid through the filter housing. After prewetting and pretreatment of the filter, if performed, the filter housing or other rigid container housing the filter is filled with a noncontaminating liquid such as ultrapure deionized water to immerse the filter. Then, the rigid container and the filter are sanitized by a suitable method, including any of the methods described above for use in sanitizing a filter within a flexible bag, such as sanitizing by heating in an autoclave.

When a filter in a rigid container, such as a filter housing, is sanitized by heating, the container may be either sealed or vented. Thus, if the walls of the container are strong enough to resist the internal pressure which develops in the container during heating of the noncontaminating liquid, the container may be completely sealed during heating by closing all the fluid ports or other openings in the container. If the container has relatively thin walls which could be damaged by the internal pressure during heating, the container may be vented by a suitable venting mechanism. Venting may be carried out through any suitable portion of the container. When the container is a filter housing, it will typically be equipped with a plurality of fluid ports, such as a fluid inlet, a fluid outlet, or an air vent, and the housing may be vented through any one or more of these fluid ports or through different opening intended specifically for use in venting during heating. The fluid ports or other openings which are not used for venting may be closed off during heating by conventional closures (pipe plugs, pipe caps, tube covers, etc.) appropriate to the structure of the individual

12

fluid ports. A vent filter, such as one of the vent filters used in the embodiments of FIGS. 1-7, may be connected to the fluid port used for venting in order to prevent contaminants from entering the container through the fluid port during heating or when the container is being cooled at the completion of heating. As in the previous embodiments, the vent filter preferably has a sterilizing grade filter medium, and the filter medium may be hydrophobic, if desired, to prevent the noncontaminating liquid from leaking from the container through the fluid port to which the vent filter is connected.

The noncontaminating liquid preferably fills the container as much as possible to exclude all free air from the container during heating. To help free air escape to the outside of the container during the introduction of the noncontaminating liquid, it may be helpful to agitate the container or to introduce the noncontaminating liquid from more than one end of the container. Alternatively, suction may be applied to a fluid port at one end of the container, and the noncontaminating liquid may be introduced through a fluid port at the other end of the container. The filter is preferably mostly immersed (at least 50% of its volume), and most preferably it is entirely immersed in the noncontaminating liquid at the start of sanitizing.

When sanitizing a filter within a rigid container which is vented, such as a vented filter housing, the level of the noncontaminating liquid within the container will usually drop due to vaporization of the noncontaminating liquid. When the container is cooled subsequent to heating, air may enter the container through the vent filter and form a pocket of air in the upper portion of the container above the surface of the noncontaminating liquid. However, as in the case when the container is a flexible bag, it is not detrimental to have some air remaining in the housing after cooling because the air will be free of microorganisms after passing through the vent filter and be at 100% relative humidity, so it will neither contaminate nor dry out the filter. Preferably, there is a sufficient amount of the noncontaminating liquid remaining in the container at the completion of cooling that the filter will be at least 50% immersed, more preferably at least 90% immersed, and still more preferably substantially 100% immersed in any attitude of the container.

A vent filter may be left connected to the container at the completion of sanitizing and shipped to the customer along with the filter package, or the vent filter may be detached and replaced by a closure to hermetically seal the container and allow the vent filter to be reused. If the vent filter is detached, the detachment is preferably performed in a manner which prevents contaminants from entering the container. If the vent filter has a hydrophobic filter medium and is left attached to the container, it is possible but not necessary to close the downstream fluid port of the vent filter, because the hydrophobic filter medium can prevent the noncontaminating liquid from leaking from the container.

FIG. 8 illustrates an embodiment of a filter package according to the present invention in which a rigid container for housing a filter 80 during sanitizing is a filter housing. The illustrated filter package comprises a disposable filter assembly available from Pall Corporation under the trademark DFA. The assembly includes a housing 90 having first and second fluid ports 91 and 92 and manually operated vents 93 and 94 which can be used to vent gas or liquid from the housing 90. The filter 80 which is disposed inside the housing 90 includes a pleated filter element 81 surrounding a hollow perforated core 82, a blind end cap 83 sealed to one end of the filter element 81, and an open end cap 84 sealed to the other end of the filter element 81 and to the second fluid port 92. It may also include an unillustrated perforated

US 6,174,439 B1

13

cage surrounding the filter element 81. The illustrated filter 80 is intended primarily for radially inward flow, so the first fluid port 91 usually serves as an inlet and the second fluid port 92 usually serves as an outlet, although the functions of the two fluid ports may be reversed. The filter 80 and the housing 90 can be made of any materials which can withstand the conditions (such as temperatures) to which they may be subjected during sanitizing. For example, the filter element 81 of the illustrated filter 80 has a PTFE filter medium, and the core 82, the end caps 83 and 84, and the housing 90 are made of polypropylene. Such a filter assembly can be sanitized by heating in an autoclave.

During sanitizing by heating in an autoclave and subsequent cooling, the housing 90 is preferably connected to a hydrophobic, sterilizing grade vent filter to enable vapor generated by heating to escape to the outside of the housing 90 while preventing microorganisms or other contaminants from entering the housing 90. The vent filter can be connected to any one or more of the fluid ports of the housing 90. In the case of the illustrated filter assembly, the housing 90 is preferably vented through at least the fluid port connected with the open end cap 84, and the open end cap 84 is preferably disposed higher than the blind end cap 83 during sanitizing so that vapor of the noncontaminating liquid 30 and other gases generated inside the core 82 of the filter 80 can flow upwards and out of the filter 80 through the open end cap 84 and not be trapped within the core 82. Fluid ports which are not vented may be sealed off during sanitizing by a stopper, a cap, or other suitable closure.

The vent filter 70 in this embodiment is a commercially available syringe filter like that illustrated in FIG. 7, but it may be any other type of vent filter, such as the types shown in FIG. 3 or FIG. 7. The illustrated vent filter 70 has two fluid ports 71 and 72, one of which 71 is formed with internal threads which can be screwed directly onto external threads formed on the second fluid port 92 of the housing 90. Instead of being connected directly to a fluid port of the housing 90, the vent filter 70 may be connected to a fluid port by a connecting member such as a threaded adapter or flexible polymeric tubing 96, as shown in FIG. 9. When tubing 96 is employed, the vent filter 70 may be equipped with a hose barb connector designed for connection to tubing, and the fluid port 92 of the housing 90 to which the vent filter 70 is to be connected may be either formed with a hose barb connector or fitted with a commercially available adapter 97 which has a hose barb connector at its outer end and which screws over the fluid port 92. Tubing 96 is a convenient means of connecting a filter housing of a filter assembly with a vent filter because at the completion of sanitizing and cooling of the filter assembly, the filter housing 90 can be hermetically sealed by heating the tubing 96 at a location (such as that shown by the dashed lines in FIG. 9) between the fluid port 92 and the vent filter 70 to melt the tubing 96 closed. The tubing 96 can be severed on the outer side of the melted portion to leave a short length of the tubing 96 attached to the housing 90, and the vent filter 70 can then be detached from the outer end of the tubing 96 and reused. Tubing 96 can be used not just with a syringe filter but with any of the other types of vent filters described above. During sanitizing, the tubing 96 may contain air, or it may be partially or completely filled with the noncontaminating liquid, so that as the noncontaminating liquid within the housing 90 is boiled off, liquid within the tubing 96 can flow into the housing 90 to replace the liquid which boiled off.

If only one of the fluid ports of a filter housing 90 is vented during sanitizing by heating, a pressure differential may develop across the filter element 81 between the side

14

communicating with the inlet 91 and the side communicating with the outlet 92. If such a pressure differential is large enough to drive vapor generated by the heating through the filter element 81, the vapor passing through the filter element 81 may result in dewetting of portions of the filter element 81. In order to prevent vapor from being driven through the filter element 81, it may be desirable to simultaneously vent the housing 90 on both the upstream and downstream sides of the filter element 81, i.e., to vent a region communicating with the inlet 91 and a region communicating with an outlet through two or more fluid ports. For example, both the inlet 91 and the outlet 92 may be simultaneously vented, or the outlet 92 and one or both of the air vents 93, 94 may be simultaneously vented. FIG. 10 schematically illustrates an embodiment in which a filter assembly is vented from both the upstream and downstream sides of a filter element. The filter assembly in this figure, only the outlet end of which is shown, is identical to the disposable filter assembly of FIG. 8. During sanitizing, the outlet 92 and the outlet-side air vent 94 are connected to a vent filter 70 in the form of a sterilizing grade syringe filter, for example, by flexible polymeric tubing 96 and a tee fitting 98 which joins the tubing 96 for the outlet 92 with the tubing 96 for the outlet-side air vent 94. The outlet 92 is equipped with a hose barb adapter 97, as in the embodiment of FIG. 9. The cap on the air vent 94 can be removed to enable the tubing 96 to be connected to the air vent 94. If desired, the air vent 94 can be fitted with a hose barb adapter similar to the one installed on the outlet 92. Sanitizing can be performed under the same conditions described for the previous embodiments. At the completion of sanitizing and cooling, the tubing 96 for both the outlet 92 and the outlet-side air vent 94 can be severed by heating the tubing 96 along the dashed lines, for example, to melt the tubing 96 closed and hermetically seal the housing 90. One or more vent filters can be connected to a plurality of fluid ports of a housing in any other desired manner. For example, a plurality of vent filters can be directly connected to the housing 90 in the manner shown in FIG. 8.

During sanitizing, the orientation of the filter housing 90 is not critical, but preferably the housing 90 is oriented as shown in FIG. 10 so that vapor can rise to the upper end of the housing 90 and be easily vented through fluid ports 92 and 94.

The vent filters shown in FIGS. 6 and 7 can also be used in a manner similar to that shown in FIG. 5 to simultaneously vent a plurality of filter packages through a single vent filter.

According to another form of the present invention, a vent filter for use during sanitizing of a filter may comprise a filter medium mounted directly on a fluid port of a filter housing or other container. FIG. 11 illustrates a portion of an embodiment of a filter package according to the present invention employing such a vent filter. The illustrated filter package is formed from a commercially available filter assembly, such as one available from Pall Corporation under the designation LDFF, although many other types of filter assemblies can also be employed. The filter assembly includes a rigid, cylindrical polymeric housing 100 equipped at one of its ends with an outlet 101 and an outlet side air vent 110, each having a hollow bore communicating with the inside of the filter housing 100. The unillustrated opposite end of the housing 100 is equipped with an inlet and an inlet side vent similar in structure to the outlet 101 and the outlet side air vent 110. An unillustrated cylindrical filter is disposed inside the housing 100 along a fluid path connecting the inlet and the outlet 101. Like the filter 80 shown in FIG. 8, the filter of the illustrated filter assembly has a blind end cap at one

US 6,174,439 B1

15

of its ends and an open end cap sealed to the outlet 101 at its other end. The air vents communicate with the interior of the housing 100 surrounding the filter. Each of the fluid ports, i.e., the inlet, the outlet 101, and the air vents can be sealed by a cap-like closure and a nut which is formed separately from the closure and secures the closure to the fluid port. For example, the outlet side air vent 110 is equipped with a blind closure 111 having an open lower end which fits over the outer end of the air vent 110. A nut 112 slides over the top of the closure 111 and engages with external threads formed on the air vent 110 to hold the closure 111 in place. The outlet 101 can be sealed by a similar, unillustrated blind closure and a nut 106 for holding the blind closure in place.

Any one or more of the fluid ports of the housing 100 may be vented during sanitizing. For the reasons given with respect to the embodiment of FIG. 8, preferably at least the outlet 101 is vented to prevent vapor from accumulating within the hollow center of the filter during sanitizing. The vent filter for the outlet 101 in this embodiment comprises a sheet of filter medium 102 mounted directly over the open outer end of the outlet 101. The filter medium 102 is not restricted to any particular type but is preferably a sterilizing grade filter medium which can prevent bacteria and other contaminants from entering the housing 100 while permitting vapor to escape from the housing 100 during sanitizing. If desired, the filter medium 102 may be hydrophobic to prevent a noncontaminating liquid with which the housing 100 is filled from leaking from the housing 100 when the housing 100 is tilted. A membrane filter medium is particularly suitable as the filter medium 102 because a membrane can be sufficiently thin and flexible to readily conform to the shape of the outlet 101 without tearing. An example of a suitable membrane filter medium is a sterilizing grade PTFE membrane. Examples of other suitable membrane materials are polyvinylidene fluoride and hydrophobic nylon. The thickness of the filter medium 102 is not limited and can be chosen based on the strength desired of it. Typically, the thickness will be in the range of 0.0254–0.127 mm. Depending upon the physical strength of the filter medium 102, it may be desirable to dispose a support member 103 which is permeable to vapor of the noncontaminating liquid and stiffer than the filter medium 102 adjacent the outer surface of the filter medium 102 to prevent the filter medium 102 from bulging outwards during sanitizing. A similar support member 103 can also be disposed adjacent the inner surface of the filter medium 102 to prevent the medium 102 from deforming inwards as well. In the present embodiment, the support member 103 comprises a thin sheet of a porous, nonwoven fluoropolymer fabric which is permeable to vapor of the noncontaminating liquid. Examples of other possible support members are a thin perforated plate, a porous woven fabric, and a porous mesh. It is generally not necessary for the support member 103 to perform any function except physically support the filter medium 102, i.e., it is not necessary for the support member 103 to remove particulates from fluid which passes through it during sanitizing, and preferably the support member 103 is sufficiently porous that it does not produce any significant pressure drop.

The filter medium 102 and the support member 103 can be of any convenient size, but preferably each has a surface area which is at least as large as the cross-sectional area of the bore in the outlet 101 so that they can completely cover the bore. They may be cut from sheets into any convenient shape.

The filter medium 102 and the support member 103 can be attached to the outlet 101 in any desired manner which

16

can prevent microorganisms and other contaminants from bypassing the filter medium 102, such as by bonding or by a mechanical connector (a ring, a hose clamp, etc.) which fits around the outlet 101 and grasps the filter medium 102. A mechanical connection is generally preferable to bonding, since bonding has the potential to damage the filter medium 101 or the housing 100 and introduce contamination. In the illustrated embodiment, after the housing 100 has been filled with a noncontaminating liquid to immerse the filter contained within the housing 100, the medium 102 and the support member 103 are placed over the top of the outlet 101 and then held in place by an open-ended, cap-like closure 104 which slides over the outer end of the outlet 101 and is retained by nut 106 which slides over the closure 104 and engages with external threads formed on the outlet 101. FIG. 15 shows the appearance of the outlet 101 during sanitizing. The filter medium 102 is sufficiently thin and flexible that it can be laid over the threads of the outlet 101 and the nut 106 can be screwed over the filter medium 102 without damage to the portion of the filter medium 102 covering the outer end of the bore of the outlet 101. The open-ended closure 104 can be initially manufactured with an open end, or it can be obtained by cutting off the blind end of a blind closure like the closure 111 for the outlet side air vent 110, or by punching perforations in the outer end of a blind closure. To make it easier to slide the open-ended closure 104 over both the filter medium 102 and the support member 103, one or more axial slits 105 may be cut in the outer wall of the closure 104 to permit radial expansion of the closure 104.

Any of the other fluid ports of the housing 100 may also be provided with a vent filter of the type employed for the outlet 101, or with any other type of vent filter.

The illustrated filter assembly can be sanitized under the same conditions described with respect to any of the previous embodiments. When the filter assembly is sanitized by heating in an autoclave, the outlet 101 is preferably elevated with respect to the rest of the housing 100 so that the vapor which is generated during heating can rise towards the outlet 101 and be readily vented from the housing 100. At the completion of cooling of the filter assembly following sanitizing, if the filter medium 102 is hydrophobic, the filter housing 100 may be shipped to the customer with the open-ended closure 104 left on the outlet 101, since the hydrophobic filter medium 102 can prevent water from leaking out of the housing 100. However, to prevent the filter medium 102 from being inadvertently punctured during handling of the filter assembly, it may be desirable to replace the open-ended closure 104 with a blind closure or other member which can protect the filter medium 102, like the blind closure 111 for the outlet side air vent 110. The open-ended closure 104 can be easily replaced by unscrewing the nut 106 from the outlet 101, removing the open-ended closure 104 without removing the filter medium 102, and then placing a blind closure over the filter medium 102. At this time, the support member 103 may be either left in place atop the filter medium 102 or removed to make it easier for the blind closure to slide over the outlet 101. The blind closure may be loosely mounted on the outlet 101, or it may be pressed tightly against the outlet 101 by the nut 106 to hermetically seal the housing 100. When a customer is ready to use the filter package, he can remove the nut 106 and the blind closure and then peel the support member 103 (if still present) and the filter medium 102 off the outlet 101. Since no bonding agent is used to attach the filter medium 102 to the outlet 101, the filter medium 102 can be easily separated from the outlet 101 without leaving any residue.

In a similar manner, a filter medium can also be mounted directly on the outer ends of the vent tubes 42 and 65 used

US 6,174,439 B1

17

in the embodiments of FIGS. 1-7 in which a container of a filter package comprises a flexible bag 20. For example, a membrane filter medium and a support member can be disposed over the outer end of a vent tube and held in place by an open-ended closure and a nut like those used in the embodiment of FIGS. 11 and 12. Similarly, a filter medium can be mounted directly on any of the fluid ports of the filter assembly shown in FIG. 8.

A vent filter comprising a filter medium mounted directly on a fluid port of a container is advantageous in that it can be readily assembled by a user from inexpensive hardware, so equipment costs are extremely low.

The present invention will be further illustrated by the following examples.

EXAMPLE 1

A rectangular sheet of PFA film measuring 6 inches×18 inches and having a thickness of 0.005 inches was folded in half and then heat sealed along two edges to obtain an elongated bag measuring 3 inches×18 inches and having one open end. A vent hole was punched in the bag near the open end using a hole punch, and a vent tube like that shown in FIG. 3 was sealingly connected to the bag at the vent hole.

A pleated filter (AB1F0013EH1 filter available from Pall Corporation under the trade designation "Super-Cheminert" and having a PTFE single-layer filter medium) was prewetted by dipping in isopropyl alcohol for 5 minutes at room temperature (approximately 25° C.). The isopropyl alcohol was then removed by flushing the filter with deionized water for at least 5 minutes. The filter was next transferred to a tank of hot deionized water at approximately 71° C. for 60 minutes to perform leaching. The filter was then placed into the bag through the open end, and this end was sealed using a heat sealer.

Ultrapure deionized water (initial resistivity of 18 MΩ·cm) was introduced into the bag through the vent tube to completely submerge the filter. A hydrophobic, sterilizing grade PFA filter (Pall Model DFA4001FRP) was then sealingly connected to the vent tube as a vent filter.

The bag was next placed into an autoclave and heated for one hour under standard sterilizing conditions of 15 psi gauge and approximately 120° C. to sterilize the contents of the bag. At the end of one hour, the bag was removed from the autoclave and cooled in air to a safe handling temperature. The bag was then hermetically sealed below the surface of the water using a heat sealer to obtain a completed filter package. At the time of sealing, the upper portion of the bag including the vent tube and the vent filter was detached from the lower portion of the bag containing the filter. The vent tube, the associated hardware, and the vent filter were detached from the upper portion of the bag for reuse, and the upper portion of the bag was discarded.

EXAMPLE 2

This example illustrates sterilizing a filter assembly like that illustrated in FIG. 8 to obtain a sterilized filter package. The filter assembly, which comprises a filter 80 and a rigid polymeric housing 90, is a disposable filter assembly available from Pall Corporation under the trademark DFA.

The filter 80 is prewetted by passing isopropyl alcohol at room temperature (approximately 25° C.) through the housing 90, the isopropyl alcohol being introduced through the inlet 91 and discharged through the outlet 92. The isopropyl alcohol is then removed by flushing the filter housing 90 with deionized water for 5 minutes. The deionized water is then allowed to drain from the housing 90.

18

The outlet-side air vent 94 is shut, the outlet 92 is closed with a threaded cap 95, and ultrapure deionized water (initial resistivity of 18 MΩ·cm) is introduced into the housing 90 through the inlet 91 with the inlet-side air vent 93 open and the housing 90 upright so that air can escape through the inlet-side air vent 93. When the ultrapure deionized water reaches the top of the inlet 91, the inlet-side air vent 93 is shut and the inlet 91 is closed with a cap 95. The housing 90 is then inverted, the outlet-side air vent 94 and the outlet 92 are opened, and additional ultrapure deionized water, if necessary, is added to the housing 90 through the outlet 92 to completely fill the housing 90 and exclude all air from the housing 90. In this state, the filter 80 is completely immersed in the ultrapure deionized water inside the housing 90. The closure for the outlet-side air vent 94 is removed, and a vent filter 60 like that shown in FIG. 6 comprising a filter holder and a hydrophobic, sterilizing grade membrane filter medium 64 is attached to both the outlet 92 and the outlet-side air vent 94 by tubing 96 and a tee fitting 98 in the manner shown in FIG. 10. The uppermost end of the tubing 96 is attached to fluid port 61a of the vent filter 60.

The filter assembly and the vent filter 60 are then placed into an autoclave and heated for one hour at 15 psi gauge and approximately 120° C. to sterilize the entire filter assembly. During sterilizing, the housing 90 is substantially upright with the outlet 92 disposed higher than the inlet 91. At the end of this time, the filter assembly and the vent filter 60 are removed from the autoclave and cooled in air to a safe handling temperature. The tubing 96 is then severed by heating the tubing 96 at a location between the tee fitting 98 and the filter assembly to hermetically seal the assembly and obtain a completed filter package. The tubing 96 can be removed from the filter assembly by the customer when he is ready to use the assembly. The vent filter 60 can be reused with the same or a different filter medium 64.

What is claimed is:

1. A filter package enabling the filter to be stored and shipped in a wet state without the filter drying out, the filter package comprising a sanitized filter cartridge for removal of particulates from a fluid and having a porous filter medium including a plurality of pores through which the fluid can pass between upstream and downstream sides of the filter cartridge during filtration to remove particulates from the fluid, a flexible bag which surrounds the filter cartridge and has walls extending between an upper end and a lower end thereof, the walls comprising a polymeric material impervious to microorganisms and liquid water, and sanitized water hermetically sealed inside the bag and substantially permeating the pores of the filter medium and immersing substantially 100% of a volume of the filter cartridge, the bag being hermetically sealed around the filter cartridge with substantially all air excluded from the bag.

2. A filter package as claimed in claim 1 wherein the filter cartridge has a bore filled with water, wherein the water comprises sterilized water, and wherein the bag comprises a translucent or transparent polymeric material.

3. A filter package as claimed in claim 2 wherein the filter cartridge comprises a pleated filter element and wherein the bag is heat sealed and substantially all gas is excluded from the bag.

4. A filter package as claimed in claim 1 wherein the sanitized water contains substantially no bactericides.

5. A filter package having a long shelf-life and containing substantially no contaminants, the filter package comprising a sanitized filter cartridge for removal of particulates from a fluid and having a porous filter medium including a plurality of pores through which the fluid can pass between an

US 6,174,439 B1

19

upstream side and a downstream side of the filter cartridge as particulates are removed from the fluid, a flexible bag hermetically sealed around the filter cartridge and having an inside surface, an outside surface, and walls comprising a material impervious to microorganisms and liquid water, and sanitized water contacting the inside surface of the bag and hermetically sealed inside the bag and substantially permeating the pores of the filter medium so that the filter cartridge will not dry during storage and shipment and immersing substantially 100% of a volume of the filter cartridge, substantially all air being excluded from the bag.

6. A filter package as claimed in claim 5 wherein the water comprises sterilized water.

7. A filter package as claimed in claim 5 wherein the filter cartridge has a central bore which is filled with water.

8. A filter package as claimed in claim 5 wherein the water comprises ultrapure water.

9. A filter package as claimed in claim 5 wherein the level of contaminants in the water is in the parts per billion range.

10. A filter package as claimed in claim 5 wherein the bag comprises a translucent or transparent polymeric material.

11. A filter package as claimed in claim 5 wherein the bag comprises a non-polymeric material.

12. A filter package as claimed in claim 5 wherein the sanitized water contains substantially no bactericides.

13. A filter package comprising a flexible bag having an inside surface, an outside surface, and walls comprising a material impervious to microorganisms and liquid water, a sanitized filter cartridge disposed in the bag and including a filter medium having pores through which a fluid can pass between a upstream side and a downstream side of the filter

20

cartridge to be filtered by the filter medium, and sanitized water hermetically sealed inside the bag and contacting the inside surface of the bag, substantially permeating the pores of the filter medium, and immersing substantially 100% of a volume of the filter cartridge.

14. A filter package as claimed in claim 13 wherein the filter has a central bore which is filled by the water.

15. A filter package as claimed in claim 14 wherein the bag has an upper end and the filter has a first lengthwise end which is open and a second lengthwise end which is farther from the upper end of the bag than the first lengthwise end.

16. A filter package as claimed in claim 14 wherein the filter comprises a pleated filter cartridge.

17. A filter package as claimed in claim 16 wherein substantially all gas is excluded from the flexible bag.

18. A filter package as claimed in claim 17 wherein the bag comprises a translucent or transparent polymeric material.

19. A filter package as claimed in claim 17 wherein the water comprises ultrapure water.

20. A filter package as claimed in claim 17 wherein the level of contaminants in the water is at most in a parts per billion range.

21. A filter package as claimed in claim 17 wherein the water comprises sterilized water.

22. A filter as claimed in claim 14 wherein the water comprises sterilized water.

23. A filter package as claimed in claim 13 wherein the sanitized water contains substantially no bactericides.

* * * * *

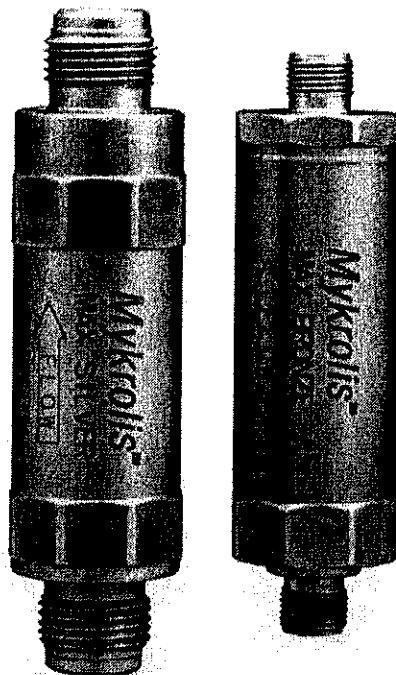
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Exhibit C



Wafergard[®] MAX In-Line Teflon[®] Gas Filters

Superior particulate filtration for ultra high purity applications



Low pressure drop filter for high purity process gas

Wafergard MAX Inline Teflon Gas Filters provide the industry's best filters for ultra-high purity gas systems. Available in Bronze and Silver performance levels, the MAX series is specifically designed for high flow and low delta-pressure applications. The Wafergard product line provides ultimate process protection. Whatever your process needs, the Wafergard family of filters provides ultimate purity, superior corrosion resistance, and excellent compatibility with all gases.

Multiple Use Application

Wafergard MAX is suitable for facility hook-up, valve manifold boxes, and point-of-use gas panel applications. The high surface area membrane provides minimal pressure drop for low vapor pressure gases. The all Teflon filter element is compatible with high purity gases as well as general nitrogen and CDA applications.

Product Features

Bronze or Silver Performance Level

All Teflon filter cartridge, high surface area PTFE membrane and PFA support structure

Small footprint

Pre-conditioned (Silver only)

Available in VCR[®], Swagelok[®] and buttweld fittings

Product Benefits

Select ultra high purity (Silver) or high purity (Bronze) surface finish

High flow, low pressure drop, excellent compatibility with inert, reactive, corrosive and oxidizer gases

Easier integration in gas panel, VMB or facility hook-up

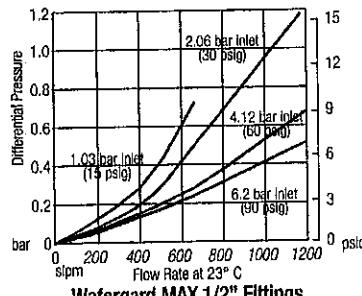
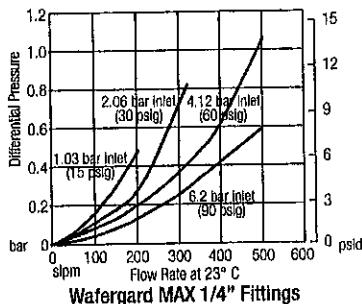
Minimal installation down time

Multiple fitting styles to provide one-source flexibility for all your gas filtration needs

Product Profile

Wafergard MAX In-Line Teflon Gas Filters - Ordering Information

Materials	Filter element: Hydrophobic Teflon PTFE membrane supported by molded Teflon PFA structures
Downstream Cleanliness	Housing: Electropolished Low Sulfur 316L stainless housing Particles: Less than 0.03 particles/Liter (<1 particle/ft ³) greater than 0.01 µm Volatile (Silver): <10 ppb H ₂ O and O ₂ . Below LDL THC (lower detection limit is 5 ppb)
Removal Rating	≥ 0.003µm
Surface Finish Interior	Bronze: ≤ 20 µin Ra Silver: ≤ 7 µin Ra
Helium Leak Rating	Qualified: 2x10 ⁻¹⁰ atm.cc/sec = 2x10 ⁻¹¹ Pa.m ³ /sec Tested: 1x10 ⁻⁹ atm.cc/min = 1x10 ⁻¹⁰ Pa.m ³ /sec
Operating Conditions	Maximum Inlet Pressure: Silver and Bronze 1/4" and 1/2" VCR compatible fittings: 4.0 MPa (40 bar, 580 psig) Silver Butt Weld and Bronze 1/4" Swagelok compatible fittings: 7.0 MPa (70 bar, 1015 psig) Bronze 1/2" and 3/8" Swagelok compatible fittings: 7.5 MPa (75 bar, 1088 psig) Maximum Forward Differential Pressure: 7 bar (102 psid) Maximum Reverse Differential Pressure: 2.5 bar (36 psid) Maximum Operating Temperature: 120°C
Warranty	Bronze: 1 year; Silver: 3 years
Particle Retention	Greater than 99.999999% removal of all particles (referenced at the most penetrating particle size).



Wafergard MAX Bronze - Ordering Information

WGMX

MB



Fittings

- RR2 = 1/4" Male VCR compatible fittings
- RR4 = 1/2" Male VCR compatible fittings
- SS2 = 1/4" Swagelok compatible fittings
- SS3 = 3/8" Swagelok compatible fittings
- SS4 = 1/2" Swagelok compatible fittings
- PRF2 = 1/4" Male VCR inlet, 1/4" Female VCR outlet compatible fittings

Wafergard MAX Silver - Ordering Information

WGMX

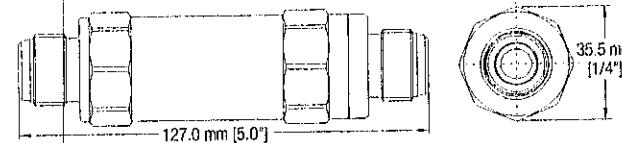
MS



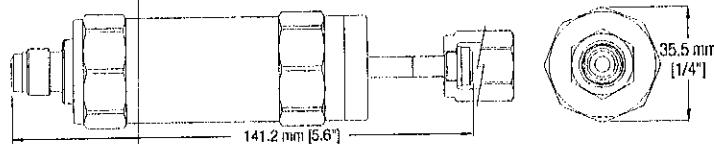
Fittings

- RR2 = 1/4" Male VCR compatible fittings
- RR4 = 1/2" Male VCR compatible fittings
- W2 = 1/4" Butt weld fittings
- W3 = 3/8" Butt weld fittings
- W4 = 1/2" Butt weld fittings

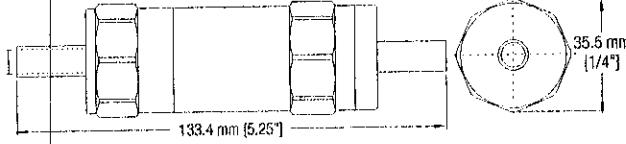
1/4" and 1/2" Gasket Seal Male Inlet/Male Outlet



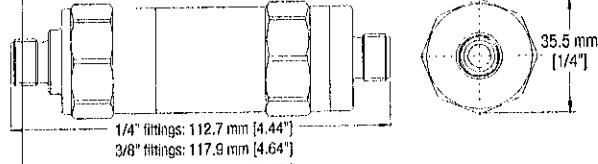
1/4" Gasket Seal Male Inlet/Female Outlet



1/4", 3/8", and 1/2" Butt Weld



Compression Seal



Consult our applications specialists with any questions.

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Allen, TX 972-359-4000

Germany 49 816 595 110

San Diego, CA 858-452-0124

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Singapore 65 6745 2422

Tokyo 81 3 5442 9718

Korea 82 31 738 5300

Kyushu 81 92 471 8133

People's Republic of China 86 21 5080 5600

Osaka 81 6 6390 0594

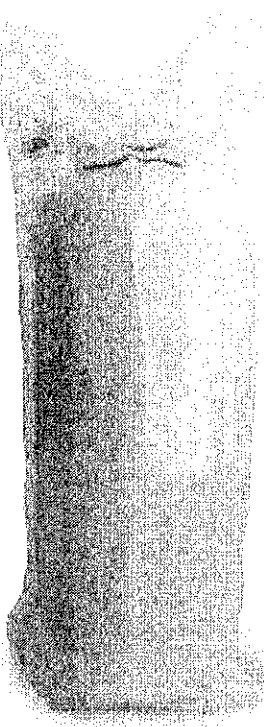
Taiwan 886 3 571 0178

Exhibit D



QuickChange[®] ATM Cartridge Filters

Advanced non-dewetting membrane technology in a high-flow, long-life prewet filter that delivers particle excursion protection, ease-of-use, rapid changeout, and safety



The Solution for Aqueous-based Chemical Processes

QuickChange filters, designed for use in any aqueous-based chemical, are shipped water-wet and require no IPA prewetting procedures. Without an IPA prewetting requirement, QuickChange filters prevent alcohol/chemical interaction, avoid potential sources of contamination, and eliminate the cost and inconvenience of hazardous waste disposal. QuickChange filters reduce filter changeout time, minimize system downtime, and lower cost-of-ownership.

Superior Filtration Efficiency and High Flow

The combination of a non-dewetting pleated membrane in a large filtration-area device allows QuickChange filters to maintain high flow, even with small pore sizes. By maintaining consistent flow rates in outgassing chemistries such as piranha, SC1 and SC2, QuickChange filters can help stabilize process conditions.

The patented QuickChange membrane offers more wettable surface, while preventing de-wetting and subsequent system downtime.

Product Features

High Membrane Surface Area

Non-dewetting PTFE membrane filter pre-wet and packaged in high-purity DI water

Unique, patented membrane

Aqueous-based chemical compatibility

Product Benefits

QuickChange ATM products incorporate 40% more membrane area within the same footprint as QuickChange ATX products, providing for high flow rate even with ultrafine particle retention.

Eliminates prewetting and flushing cycles. Eliminates chemical usage while reducing system downtime during filter changeouts. Saves time, greatly increases equipment uptime.

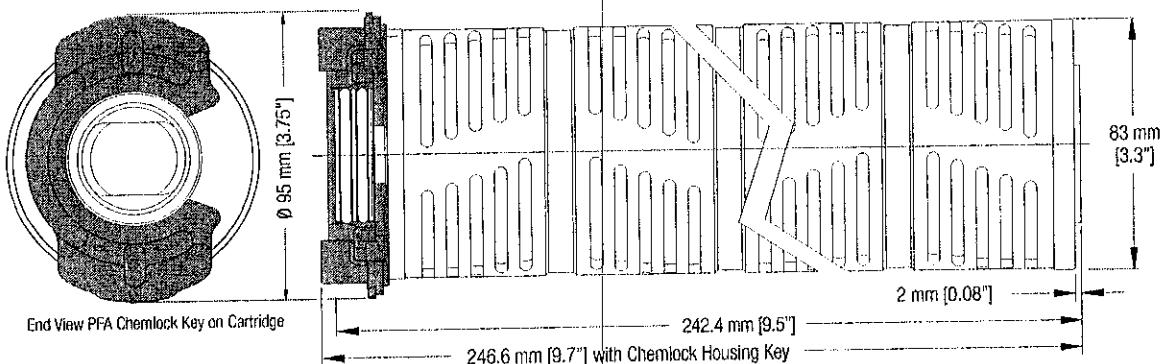
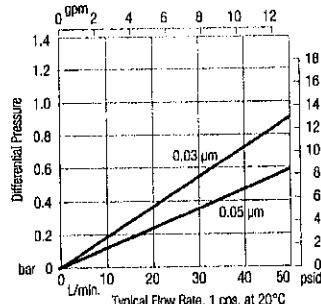
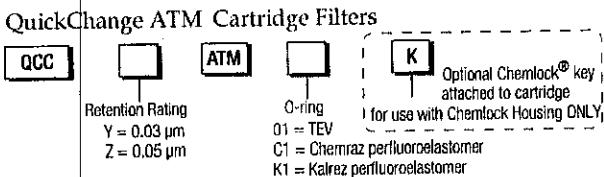
The prewet, high performance membrane provides a surface which prevents dewetting in air, during installation or chemical dumps/drains, and outgassing chemistries.

Recommended for aqueous-based chemicals at ambient temperatures (including H₂SO₄, H₃PO₄, HNO₃, HF, HCl, BOE, NH₄F, H₂O₂, TMAH, NH₄OH, and ozonated water) and elevated temperature applications (including SC1, SC2, piranha etch, nitride etch, and metal etch).

Product Profile

QuickChange ATM Cartridge Filters - Ordering Information

Materials	Membrane: Prewet, non-dewetting PTFE Supports: PFA Cartridge O-rings: Teflon fluoropolymer encapsulated Viton® fluoroelastomer O-ring (TEV), Chemraz® or Kalrez® perfluoroelastomers
Retention Rating	0.03 µm, 0.05 µm
Membrane Area	18,000 cm ² (19.4 ft ²)
Maximum Operating Conditions	Maximum Operating Pressure: 0.58 MPa (5.8 bar; 84 psi) @ 25° C 0.17 MPa (1.7 bar; 25 psi) @ 180° C Maximum Forward Differential Pressure: 0.44 MPa (4.41 bar; 64 psid) @ 25° C 0.05 MPa (0.51 bar; 7.5 psi) @ 180° C Maximum Reverse Differential Pressure: 0.35 MPa (3.5 bar; 50 psid) @ 25° C Maximum Operating Temperature: 180° C (at the above conditions)
Compatibility	Mykrolis recommends QuickChange Disposable filters for use with aqueous-based chemicals at ambient temperatures (including H ₂ SO ₄ , H ₂ PO ₄ , HNO ₃ , HF, HCl, BOE, NH ₄ F, H ₂ O ₂ , TMAH, NH ₄ OH, and ozonated water) and elevated temperature applications (including SC1, SC2, piranha etch, and metal etch).
Metallic Extractables	Guaranteed levels to < 25 µg/device (17 ppb in 1.5L 10% HCl)



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